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 i, 897.
 White, C. P., ii, 968, 972.
 Wibaut, J. P. See A. Smits.
 Widemann, M., ii, 984.
 Willgerodt, C., and R. Garti
 i, 876.
 Willstätter, R., and J. Picc
 i, 915.
 Windaus, A., and A. Wels
 i, 903.
 Witt, O. N., and E. Witte, i, 874.
 Witte, E. See O. N. Witt.

Y.

Yamamoto, T., ii, 928.
 Yates, J. See R. H. Pickard.

Z.

Zelinsky, N. D., i, 864.
 Zelinsky, N. D., and E.
 Prschevalsky, i, 845.
 Zickendraht, H., ii, 910.

ERRATA.

DECENNIAL INDEX, 1873—1882 (AUTHORS).

Line Col.

11 i for "773" read "173."

VOL. XCII (ABSTR., 1907).

PART I.

Line
9 for "hexahydro-*m*-toluic acid" read
"1-methylcyclohexane-2-carboxylic acid."

VOL. XCIII (TRANS., 1908).

1 13 for "pale yellow needles" read "almost colourless needles with only
a slight yellow tinge."
1 5* } ,, "pale yellow crystals" read "almost colourless crystals with only
5 11 } a slight yellow tinge."

VOL. XCIV (ABSTR., 1908).

PART I.

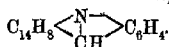
4 10* for " HCl_2Cl " read " CHCl_2 "
1 20, 21 ,, "crystallising" read "the sodium salt crystallises."
2 21 ,, $\begin{array}{c} \text{CH}_2\text{CO} \\ | \\ \text{CH}_2\text{CO} \end{array} \text{NH} \cdot \text{C}_6\text{H}_4(\text{CO}_2\text{Me})_2$ read $\begin{array}{c} \text{CH}_2\text{CO} \\ | \\ \text{CH}_2\text{CO} \end{array} \text{N} \cdot \text{C}_6\text{H}_4(\text{CO}_2\text{Me})_2$
5 17 delete "FUSIA."
3 7* for " α -Bromoisohexyl- β -aminobutyric acid" read
"a-Bromoisohexyl- β -aminobutyric acid."
9 1* }
9 13 }
9 17 } ,, "dl-fenchene" read "D-l-fenchene."
9 23 }
11 1 }
10 4 }
10 5 }
10 6 } ,, "dl-hydroxyfenchenic acid" read "D-l-hydroxyfenchenic acid."
10 9 }
11 9 }
10 12* ,, "dl-fenchene" read "D-l-fenchene."

PART II.

3 2* for "1908" read "1898."

* From bottom.

Condensation of Phenanthraquinone and o-Nitrobenzyl Chloride in presence of Stannous Chloride: Phenophenanthracridine,



Four grams of phenanthraquinone were suspended in about 50 c.c. of boiling methylated spirit containing 30 grams of stannous chloride in solution. Twenty c.c. of concentrated hydrochloric acid were gradually added, and the liquid was boiled until no insoluble residue was left. During the latter process the solution became almost black. Three grams of *o*-nitrobenzyl chloride were added carefully in very small portions, and the heating was continued on the water-bath for five or ten minutes. Even at this temperature a dark brown solid separated. When cold, the mixture was filtered, and the solid residue was washed with methylated spirit and then once with a little cold acetone. The solid was then treated with boiling concentrated aqueous potassium stannous chloride, which produced a yellow colour. Cold water was added, and the alkaline liquid was filtered off. The residue was washed with cold water, and with a little acetone, to remove black impurity still remaining. About 5 grams of a yellow powder were obtained in this way. The substance was recrystallized from benzene, from which it separated in bright yellow needles, melting at 204°. Analyses showed that it was not quite pure, but, on distilling it under diminished pressure and recrystallizing again from benzene, satisfactory results were obtained. The melting point remained unaltered. Solutions in benzene, toluene, and glacial acetic acid are fluorescent:

0.1317 gave 0.4350 CO₂ and 0.0581 H₂O. C=90.08; H=4.90.

C₂₁H₁₃N requires C=90.32; H=4.66 per cent.

The compound is therefore a *phenophenanthracridine*.

4. Preparation of 2-Aminofluorene.

Kelcey prepared 2-aminofluorene (*Ber.*, 1901, **34**, 1759) by nitration of fluorene and reducing the nitro-compound with zinc dust and stannous chloride, but it is more convenient to reduce the nitrofluorene with stannous chloride. Ten grams of finely-ground nitrofluorene were suspended in 100 c.c. of methylated spirit. Twenty-five grams of stannous chloride were added, and the mixture was boiled under a reflux condenser until a clear solution was obtained, after which about 25 c.c. of concentrated hydrochloric acid were gradually added, and the boiling continued for about

The addition of hydrochloric acid caused a white

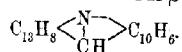
precipitate to form, which, however, slowly dissolved. On cooling the liquid, a white double salt separated. This was collected with excess of boiling aqueous sodium hydroxide, and the liquid again collected. The residue was extracted two or three times with boiling methylated spirit, and filtered from an insoluble residue. On cooling the filtrates, white needles of 2-aminofluorene, melt at 129°, separated. They could be further purified by recrystallisation from benzene.

5. Condensation of 2-Aminofluorene with Methylene Diiodide

2-Aminofluorene reacted with methylene dichloride in a sea tube heated to 250°. A non-crystallisable, brown powder was obtained, which did not melt below 300°, and which could not be purified by recrystallisation nor by distillation. Methylene diiodide, however, reacts very energetically with 2-aminofluorene when heated. The two substances were warmed together in a test tube immersed in an oil-bath, when it was found that the reaction began at about 120°. The contents of the tube were treated in the same manner as in the case of the corresponding experiment with 9-aminophenanthrene, but the product could not be purified. On distillation under reduced pressure, it decomposed, and a suitable solvent could be found from which to recrystallise it. It was therefore not analysed. It was insoluble in water, alcohol, light petroleum; sparingly soluble in boiling acetone or in boiling ethyl acetate, with slight fluorescence, and readily so in benzene, toluene or cold pyridine.

6. Condensation of a Mixture of 2-Aminofluorene and α -Naphthol with

Methylene Di-iodide: Fluorene- $\overset{\text{N-}\alpha}{\underset{\text{CH-}\beta}{|}}$ -naphthacridine,



This reaction is much more easily controlled than that between methylene di-iodide and 2-aminofluorene alone. Equimolecular proportions of α -naphthol and 2-aminofluorene were heated together in an open test-tube in an oil-bath. One molecular portion of methylene di-iodide was then added, when it was found that the contents of the tube solidified at about 148°. As soon as this occurred, heating was discontinued, and the contents of the tube were treated with boiling methylated spirit until almost the whole of the substance was dissolved. Excess of aqueous potassium hydroxide was added to the solution, when a solid separated. The latter was collected, washed with water and acetone, dried

stilled under reduced pressure, and recrystallised two or three times from toluene. Yellow needles, melting at 259° , were obtained, which, on analysis, proved to be *fluorene*- $\overset{\text{N-}\alpha}{\text{CH-}\beta}$ -*naphthacridine*. Great care had to be taken during the analysis to avoid an incomplete combustion of the carbon:

0.1076 gave 0.3584 CO_2 and 0.0495 H_2O . $\text{C}=90.84$; $\text{H}=5.11$.
 $\text{C}_{24}\text{H}_{15}\text{N}$ requires $\text{C}=90.85$; $\text{H}=4.73$ per cent.

In conclusion, I desire to express my thanks to Prof. Collie and Assistant-Prof. Smiles for much valuable advice during the course of this investigation.

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 UNIVERSITY COLLEGE,
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CLXXII.—*The Solubility of Lime in Water.*

by GERALD TATTERSALL MOODY and LEWIS THOMAS LEYSON.

The observations described in this paper were the outcome of an attempt to adapt for use in volumetric analysis a solution of lime saturated at a known temperature, in the anticipation that this would be of constant strength. When, however, reference was made to published tables, it was found that different observers of the solubility of lime in water had recorded widely divergent numbers and that in no case had a satisfactory method of determining solubility been used.

In a preliminary series of experiments, several solutions were prepared by slaking recently-burnt samples of best commercial white lime and shaking each separately with water during three hours at 18° . Although prepared under exactly similar conditions, when these were separately titrated with a decinormal solution of hydrogen chloride it was found that they were very unequal in acid neutralising power.

In the most complete investigations on the solubility of lime are those of Lamy (*Compt. rend.*, 1878, **90**, 333) and Maben (*Pharm.*, 1883, **14**, 505). Lamy experimented with lime prepared by different processes and found that the solubility varied with the method of preparation. Maben, with knowledge of the effective methods of earlier investigators, worked under conditions

involving serious errors. Of some of these he was aware, although he took no steps to eliminate them. The errors were due mainly to defective methods of saturation, of heating and of filtration.

At an early stage it appeared desirable to check the statements made by Lamy, that lime varies in solubility according to the method adopted in preparing it. To this end, samples of lime were obtained: (1) By heating the best white marble; (2) by heating the purest calcite; (3) by heating white chalk (upper chalk, unpurified); and (4) by precipitating a solution of calcium chloride with sodium hydroxide. A large excess of each sample was separately added to water contained in a Jena flask, the flasks were placed in an incubator at 25° and vigorously shaken from time to time during ten hours. After standing, the clear solutions were removed with pipettes and separately titrated with standard acid. It was found that a volume of acid capable of neutralising 100 c.c. of lime-water prepared from calcite was able to neutralise 92.2 c.c. of lime-water prepared from white chalk, 100.9 c.c. of lime-water prepared from precipitated lime and 99.09 c.c. of lime-water prepared from marble. These remarkable variations in the degree of solubility of the samples of lime were not accidental, as they persisted even after the solutions were left in contact with the samples during several weeks. They are explained by the existence of impurities in the samples of lime obtained from material other than calcite. The lime obtained by precipitating a solution of calcium chloride was found to contain an appreciable quantity of chlorine (basically chloride?) even after as many as twenty washings with water; in fact, it was found to be impossible to remove all the chlorine by washing with water. The presence of this impurity is probably responsible for a reduction in the solubility of lime prepared by precipitation. The greater strength of the lime-water prepared from white chalk was particularly noteworthy. It appears reasonable to suppose that this might be due to the presence in solution of other substances which are capable of neutralising the acid. White chalk may contain minute quantities of alkalis other than lime and silica is known to be distributed to a greater or lesser extent through its mass. On igniting such chalk, soluble silicates might be formed and pass into solution with the lime; these would produce their own effect in neutralising acid.

To determine the effect of silica, a mixture of 20 grams of finely powdered calcite with 0.75 gram of precipitated silica was heated in a muffle furnace during three and a-half hours. The resulting lime having been dissolved in the manner already described, it was found that 92.25 c.c. of the lime-water were equivalent to 100 c.c. of lime-water prepared from calcite. In a second experi-

ent, 20 grams of calcite were heated with 0.25 gram of precipitated silica. In this case 93.1 c.c. of lime-water were found to be equivalent to 100 c.c. of lime-water prepared from calcite.

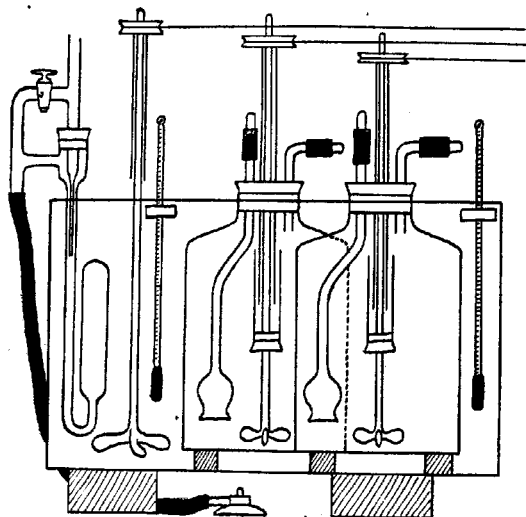
It appears, therefore, that the observed difference in the solubility of lime obtained from different sources is not ascribable to any peculiarity of the lime itself but that it is due to the presence of impurities which either affect the solubility of the lime or are capable of neutralising acids, thereby causing an error whenever lime present in solution is estimated by titration with acid. We have further found: (1) That errors may also arise in determining the solubility of lime owing to the slowness with which water is saturated with lime, even although the lime be present in great excess; (2) that saturated lime-water becomes supersaturated on its temperature is raised and that it gives up the excess of lime with extreme slowness; (3) that solutions of lime are appreciably weakened when passed through filter papers or other fibrous substances; (4) that precipitated lime containing chlorine, after strong ignition, whereby the chloride is volatilised, gives a lime-water practically identical in strength with that prepared from calcite; and (5) that lime prepared by heating calcium carbonate, obtained by precipitation with sodium carbonate, gives a lime-water which is very slightly stronger than that prepared from calcite, a fact due to the carrying down of a minute quantity of sodium carbonate with the precipitate.

On plotting Maben's numbers for the solubility of lime between 50° and 75°, a remarkable periodic curve is obtained, the solubility rising and falling as temperatures decreasing more rapidly through each alternate five degrees. It seemed in a high degree unlikely that such a curve could express the actual solubility and we therefore proceeded to devise a method by which the solubility could be determined accurately.

The lime used by us in the exact determinations was in all cases prepared by igniting the purest specimens of calcite obtainable. The ignition was conducted in a platinum dish, which was placed in a muffle furnace. In all cases in which the resulting lime was coloured, the material was rejected. The lime obtained from 150 grams of calcite was placed in a wide-mouthed bottle having a capacity of approximately three litres, which was immediately filled with recently-boiled distilled water. The mouth of the bottle was closed with a tightly-fitting rubber stopper, through which passed three tubes. The central tube formed the bearing of a glass rod, which passed to near the bottom of the bottle, where it terminated in a glass screw. The upper end of the glass rod carried a pulley connected by a belt to a revolving shaft; a "mercury trap" was

attached to the rod to guard against the admission of air or egress of solution during stirring. The second tube from which the saturated lime-water was delivered, also passed to near the bottom of the bottle and terminated in a thistle-head, which formed a filter. The upper part of the tube was bent at right angles and end attached, if necessary, to a condenser. The filtering material consisted of a layer of cotton-wool contained between filter paper strengthened on both sides by layers of fine calico, the whole being tightly and firmly attached to the rim of the thistle-head. In this arrangement, the filter was kept in the lime solution; the fil-

FIG. 1.

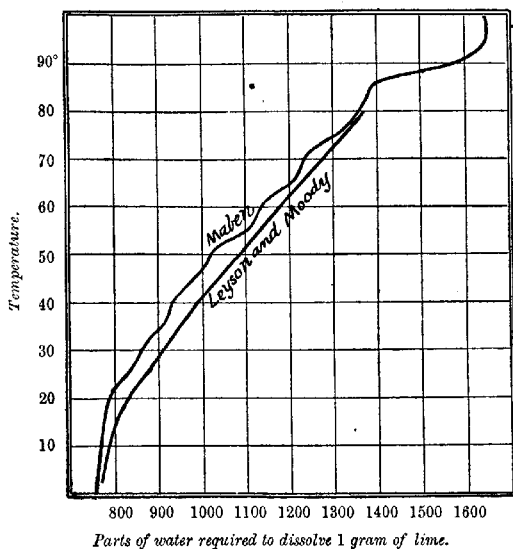


soon absorbed as much lime as it was capable of holding and an error due to filtration was obviated. The third tube in the stopper was that by which air, previously passed through a potash tower, was forced, under pressure, into the bottle, thereby allowing an required weight of solution to be driven through the filtering tube. Two of the vessels described were used and these were placed in a water-bath, the temperature of which was controlled by a thermostat (Fig. 1). Independent determinations of solubility were made from the lime-water in each bottle. The estimation of the lime in solution was not made until after ten days' stirring at a constant

perature and no result was regarded as trustworthy unless the solution, on being stirred and heated for a further twenty-four hours, showed no change in strength.

The method of estimation consisted in placing in a stoppered flask a known volume of an exactly decinormal solution of hydrogen chloride, prepared by a method described by one of us (Moody, *ibid.*, 1898, **73**, 658). The flask and contents were weighed and a quantity of lime-water, insufficient to neutralise the acid, was added into the flask, which was again weighed. The excess of acid

FIG. 2.



is then found by titration with a decinormal solution of sodium carbonate.

When working at temperatures above 30°, a short but efficient condenser was attached to the tube, from which the lime-water is delivered into the flask containing the standard acid. By this means evaporation from the lime solution during transfer was prevented and constant results were obtained for higher temperatures. The following table shows the solubilities as directly determined; column I gives the temperature and column II gives the weight

of water in grams required to dissolve 1 gram of lime (CaO) the corresponding temperature:

I.	II.	I.	II.
2°	768.5	40°	989.1
10	786.8	50	1083.0
15	804.3	60	1179.0
20	826.4	70	1274.8
25	868.7	80	1368.1
30	908.2		

It will be observed that the solubilities here recorded are appreciably smaller than those given by Maben and Lamy. For temperatures between 25° and 80°, the curve of solubility, as shown in Fig. 2, is practically a straight line, the small variation being possibly due to the dissolution of some of the material of bottles in which the solutions were made.

CHEMICAL DEPARTMENT,
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CLXXXIII.—The Chlorination of *para*-Nitroaniline.

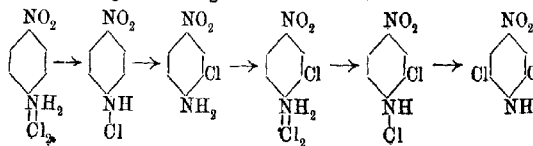
By BERNHARD FLÜRSCHHEIM.

IN a previous investigation (Trans., 1908, '93, 1463), 2:6-dichloronitroaniline was required for the preparation of 3:5-dichloro-4-bromonitrobenzene. No really satisfactory method for the preparation of the former compound being known, it was desirable to discover one, and, since it was found that dichloronitroaniline may be diazotised with concentrated sulphuric acid, when it will combine with azo-compounds such as β -naphthol, yielding the corresponding azo-dyes, it may be useful to describe the new method for its preparation.

Körner (*Gazzetta*, 1874, 4, 376) and Witt (*Ber.*, 1875, 8, 1419, 1903, 36, 4390) attempted to introduce two atoms of chlorine into *p*-nitroaniline by using chlorine gas or its aqueous solution, but generally obtained a resinous mass. Witt therefore employed potassium chlorate and hydrochloric acid as a chlorinating agent, but his method also gives an imperfect yield of an impure product.

A theoretical consideration of the process of chlorination, however, at once explained the above, and indicated how the experiment should be carried out.

The following are the stages of the reaction:



formation therefore inhibits the reaction, the rate of which
 rises with the hydrolysis of the salt, until it may result in partial
 reaction, perhaps through a local excess of chlorine.* On the other
 hand the intermediate mono-*o*-chloro-derivative unites with chlorine
 at a slower rate than does *p*-nitroaniline, both on account of its
 reduced basicity and of steric hindrance. Hence it is only after the
 aniline has been transformed into the monochloro-derivative that
 it can, to any great extent, be chlorinated further, so that it
 is precipitated and thus, under suitable conditions, escape ulterior
 reaction (compare its preparation according to D.R.-P. 109189 of
 Ila & Co.).

combining, however, a strong acid and a low temperature to
 accelerate the reaction, with a good solvent for monochloronitroaniline,
 the successive conversion of nitroaniline into dichloronitroaniline was to
 be expected, and experiment confirmed this.

Nitroaniline, being considerably more basic than its para-
 isomer, reacts much more energetically with chlorine, and its salts
 are readily hydrolysed. It follows that in high concentrations of
 acid wherein *p*-nitroaniline is still readily chlorinated, *m*-nitroaniline
 reacts, whilst in very dilute acid, *m*-nitroaniline is destroyed to a
 higher degree than *p*-nitroaniline.

EXPERIMENTAL.

Chlorination in Dilute Aqueous Acid.—Some decomposition invariably
 takes place, and the pure compounds can only be obtained by
 crystallisation.

Mono- and di-chloronitroaniline were separated by means of boiling
 hydrochloric acid, in which the latter compound is insoluble.

(1) On passing a moderate current of chlorine, at the ordinary
 temperature, through a solution of 2 grams of *p*-nitroaniline in 10 c.c.
 of concentrated hydrochloric acid and 60 c.c. of water, the precipitate
 consisted of 0.5 gram of the monochloro- and 1.1 grams of the dichloro-
 derivative.

(2) On passing a rapid current of chlorine through a boiling solution
 of 9 grams of *p*-nitroaniline in 10 c.c. of concentrated hydrochloric
 acid and 500 c.c. of water, the precipitate contained 11.2 grams of the
 mono- and 1.6 grams of the monochloro-derivative.

(3) When the current of chlorine was interrupted after one
 molecular proportion had been absorbed, the conditions being other-
 wise the same as under (2), the solution retained only a very small
 amount of nitroaniline, whereas the precipitate, besides very little

Körner (*loc. cit.*) observed the formation of tetrachloro-*p*-benzoquinone in this
 case.

dichloronitroaniline, consisted almost exclusively of monochloronitroaniline.

(4) If monochloronitroaniline is dissolved in a boiling mixture of one part of concentrated hydrochloric acid and six parts of water, the solution cooled and stirred, a current of chlorine, introduced into the suspension at the ordinary temperature, will entirely transform the monochloro- into the dichloro-compound; the same effect is produced if an excess of chlorine is passed through a boiling solution or suspension of monochloronitroaniline in one part of concentrated hydrochloric acid and 50 parts of water.

Chlorination in the Absence of Mineral Acids.—(1) In boiling water with an excess of sodium acetate, *p*-nitroaniline behaves as above; the percentage of tarry matter formed is higher.

(2) In acetic acid, both at the ordinary temperature and in a concentrated mixture, the amount of tarry matter is also considerable.

(3) In benzene, chlorination is incomplete, a great proportion of mononitroaniline being withdrawn from the reaction in the form of insoluble hydrochloride.

Chlorination in a Mixture of Acetic and Mineral Acids.—One part of *p*-nitroaniline is dissolved in three parts of acetic acid and six parts (by weight) of concentrated hydrochloric acid; the solution is cooled in ice and continually stirred while excess of chlorine is introduced. The filtered precipitate is pure dichloronitroaniline; the yield is theoretical, and the solvent can be used again for the same purpose. No monochloro-derivative separates, as it is very soluble in the mixture used.

In a previous paper (*loc. cit.*), a reaction of *m*-dinitrobenzene was described which consists in the production of a violet coloration by addition of alkali and stannous chloride to the warm aqueous solution. Dr. J. J. Blanksma has drawn the author's attention to a similar reaction of *o*-dinitrobenzene (in alcoholic solution) described by Meisenheimer (*Ber.*, 1903, **36**, 4174; 1906, **39**, 2526); Dr. Blanksma also suggested that traces of the ortho-compound, which adhere tenaciously, might cause the reaction shown by the meta-derivative. Accordingly, a "pure" commercial dinitrobenzene was recrystallized from alcohol, when it had the correct melting point (90—91°), but still showed the coloration, even in a very dilute solution. On further recrystallisation from alcohol, the melting point remained unaltered but the coloration had practically disappeared.

The reaction is therefore identical with Meisenheimer's, and is characteristic for *o*-dinitrobenzene in a dilution of one to some millions; it is consequently characteristic for technical, and even for considerably purified, *m*-dinitrobenzene in a dilution of one to many thousands. It may therefore serve for determining the presence of comme

benzene in other nitro-compounds. Commercial trinitro- and pure trinitrobenzene, for instance, are coloured red by alkali to their warm aqueous solutions, and stannous chloride this to a brownish-yellow colour, which, in the presence of benzene, becomes brownish-violet. Similarly, pure dinitro- in warm aqueous alkali is coloured pale yellow by stannous, but brownish-violet in presence of dinitrobenzene. The test also be useful in establishing the presence of traces of benzene, transforming it into its dinitro-compound.

Dr. Blanksma's suggestion, I may also point out that a test for reducing sugars, using *m*-dinitrobenzene in solution, recently described by Chavassieu and Morel (*rend.*, 1906, 143, 966), is similarly due to the presence of benzene. If a quantitative colorimetric determination of the test is to be carried out according to these authors, it is necessary to use either pure *o*-dinitrobenzene, or always the same preparation of dinitrobenzene, since the percentage of the ortho-compound in the latter is variable.

ET, HANTS.

IV.—*The Direct Action of Radium on Copper and Gold.*

By EDGAR PHILIP PERMAN.

From the observation of Sir William Ramsay, that lithium is dissolved when the emanation of radium is allowed to act on a pure salt, I thought it would be interesting and instructive to see the effect on a copper salt of the direct action of radium. For this purpose, 5 milligrams of radium barium bromide, containing 0.5 milligrams of radium bromide, were dissolved in a few drops of water and evaporated to dryness in a capsule of silica ware 1.7 cm. deep and 3 cm. in diameter. The radium salt thus formed a layer at the bottom of the capsule. About 0.5 gram of pure copper nitrate was dissolved, and treated in the same way in another capsule of the same size; the capsules were placed facing, and in contact with, one another in a wide glass tube, which was then drawn off, exhausted to 0.1 mm., and sealed. In this way it was thought that the greatest possible effect of the radium radiations would be obtained. The great activity of the radium emanation is caused presumably by the rapidity with which it throws out α -rays; but this activity decays rapidly, so that by the direct action of radium and the stored up

emanation a more economical use of the energy is made. The nitrate was made by passing oxides of nitrogen (from copper nitric acid) through a silica tube into a silica flask containing and some electrolytic copper foil. The water was specially dried and kept from contact with glass.

The copper nitrate prepared in this way was tested spectroscopically after precipitating the copper with hydrogen sulphide and evaporating to a very small bulk; traces of potassium and sodium were found, but no lithium. It was tested also by igniting in a platinum basin and extracting with water; the result was the same.

The radium and the copper were left together for three months when the tube was opened and the copper nitrate tested by the method just-described; no lithium was found.

In another experiment, 5 milligrams of radium bromide of strength 1,800,000 were allowed to act on copper sulphate in a similar way for four months. The copper salt was prepared by repeatedly recrystallising the best quality commercial copper sulphate; it could be freed completely from potassium and sodium even when crystallised in silica vessels, but no lithium could be found. After the action of radium, the spectroscopic examination gave the same result.

It may be objected that the quantity of radium employed in these experiments was too small. It must, however, be remembered that 0.000,001 milligram of lithium can be detected with the spectroscopic method, and this was confirmed with the instrument used, a spectrovision Hilger. Consequently, we can say from the second experiment that radium bromide will not convert the one-hundredth part of its weight of copper into lithium per day when acting under the most favourable circumstances. (This is allowing 0.000,001 milligram of lithium as the smallest detectable quantity.)

The Direct Action of Radium on Gold.—Assuming that the atom is broken down by the radium emanation, it was thought that the atom of gold would be still more likely to suffer disintegration. This was first tried in solution. One gram of gold chlorid dissolved in 5 c.c. of water in a weighing bottle, and 1 gram of radium barium bromide containing 1 per cent. of radium bromide was added to the same solution, which was allowed to stand for four months. The gold was then precipitated together with the radium and barium by the addition of sulphurous acid; the mixture filtered off, and the filtrate evaporated down and tested spectroscopically. The sodium line was very strong; there were traces of potassium and calcium, but no lithium could be found.

The precipitate on the filter paper, consisting of radium bromide sulphate and of metallic gold, was very thoroughly washed with water, allowed to stand, protected from dust, for three months. It was

and the washings tested again; nothing was found but a sodium. After standing again for four months, it was in the same way, with the same result. From this experiment, we said that radium bromide (under the conditions of the experiment) will not convert the one two-hundred millionth part of it of gold into lithium per day.

He has to thank Mr. R. H. Greaves, B.Sc., for valuable assistance in this work, especially in confirming all the spectroscopic observations.

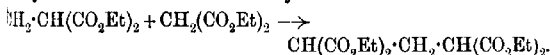
UNIVERSITY COLLEGE,
CARDIFF.

IV.—Syntheses with the Aid of Monochloromethyl Ether. Part I. The Action of Monochloromethyl Ether on the Sodium Derivatives of Ethyl Malonate and Ethyl isopropylmalonate.

By JOHN LIONEL SIMONSEN.

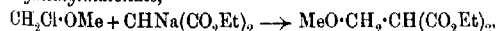
This investigation was originally commenced with the object of finding a simple method for the preparation of ethyl β -bromomethylate, $\text{CH}_2\text{Br}\cdot\text{CH}(\text{CO}_2\text{Et})_2$, since it seemed likely that this substance might be used instead of ethyl β -iodopropionate, but would have the additional advantage of containing a hydrogen atom replaceable by sodium, and thus be valuable for further syntheses.

The action of monochloromethyl ether on the sodium derivative of ethyl malonate has already been investigated by Kleber (*Annalen*, 246, 97), who isolated from the product ethyl dimethoxymalonate and ethyl dicarboxyglutarate; the formation of the latter substance he rightly interpreted as being due to the reaction of one molecule of ethyl β -methoxymethylmalonate and ethyl malonate with loss of methyl alcohol:



The present author, working probably under other conditions, has obtained a somewhat different result.

When monochloromethyl ether and ethyl sodiomalonate are condensed together under the conditions described in the experimental part of this paper (p. 1780), the main product of the reaction is *ethyl dimethoxymalonate*,



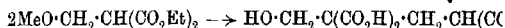
but a small quantity of ethyl dicarboxyglutarate is also formed.

That the main product of the reaction consists of ethyl β -methylmalonate, and not of ethyl dimethoxydimethylmalonate obtained by Kleber, was shown by the fact that, on treatment with hydrogen bromide, it yielded *ethyl β -bromomethylmalonate*, which on reduction and hydrolysis, gave methylmalonic acid,

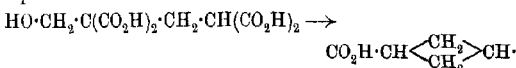


When ethyl β -methoxymethylmalonate is digested with hydrochloric acid, it yields *cis-cyclobutane-1:3-dicarboxylic acid*, and the method was to obtain evidence of the method of formation of this acid.

When ethyl β -methoxymethylmalonate is hydrolysed with hydroxide, a curious condensation takes place, with the formation of an acid, melting at 181° , which evidently consists of δ -hydroxy- α,γ,γ -tetracarboxylic acid, and has therefore been formed by the condensation of two molecules of the ester with loss of methyl alcohol:

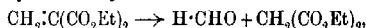


When this acid is boiled with hydrochloric acid and subsequently distilled, it loses carbon dioxide and water, and is converted into *cyclobutane-1:3-dicarboxylic acid*, a process which may be represented thus:



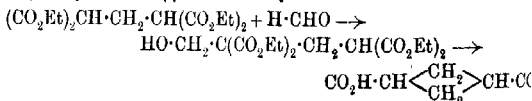
The above synthesis of *cyclobutane-1:3-dicarboxylic acid* is in some light on the mechanism of its formation from ethyl methylmalonate by hydrolysis, and seems to show that this acid is formed by the spontaneous polymerisation of methylenemalonate as was formerly thought (Bottomley and Perkin, *Trans.*, 1900, 298). The true explanation of this remarkable synthesis of a butane ring is probably the following:

A molecule of ethyl methylenemalonate is first of all decomposed into formaldehyde and ethyl malonate:



and the ethyl malonate thus formed condenses with a further quantity of ethyl methylenemalonate to give ethyl dicarboxylglutarate:

$\text{CH}_2(\text{CO}_2\text{Et})_2 + \text{CH}_2 \cdot \text{C}(\text{CO}_2\text{Et})_2 \rightarrow (\text{CO}_2\text{Et})_2\text{CH} \cdot \text{CH}_2 \cdot \text{CH}(\text{CO}_2\text{Et})_2$ which then combines with the formaldehyde present to form δ -hydroxybutane- α,γ,γ -tetracarboxylate:



and this subsequently loses water and carbon dioxide with the formation of *cyclobutane-1:3-dicarboxylic acid*.

this view is correct receives further confirmation from the fact that when ethyl β -methoxymethylmalonate is heated with acetic acid and zinc chloride, two molecules condense with loss of alcohol to form *ethyl 8-methoxybutane- α,γ,γ -tetracarboxylate*,

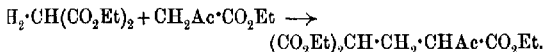


ester, when hydrolysed with hydrochloric acid, yields *cis-cyclo-3-dicarboxylic acid* (see p. 1785). The ease with which it is formed is somewhat remarkable, in view of the fact that formation of *cyclobutane* derivatives is generally attended with difficulty, as has been emphasised in a previous paper (Perkin and *Trans.*, 1907, **91**, 1739).

The tendency of ethyl β -methoxymethylmalonate to undergo condensation with loss of methyl alcohol is very marked. It has already been mentioned that one of the products of the condensation of monochloromethyl ether and ethyl malonate is ethyl dicarboxylglutarate, and the explanation of its formation suggested by Kleber is given on p. 1784.

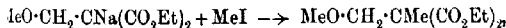
That this explanation is correct seems to be proved by the fact that ethyl malonate and ethyl β -methoxymethylmalonate condense with extraordinary ease, since it was observed (p. 1784) that, in the mixture of the two is distilled under the ordinary pressure, methyl alcohol takes place and ethyl dicarboxylglutarate is formed.

The type of reaction seems to be a general one, since ethyl β -methoxymethylmalonate readily condenses with ethyl acetoacetate in the presence of acetic anhydride and zinc chloride with the formation of *carboxy- α -acetylglutarate*,



His method of producing the same substance, which throws light on the mechanism of the above reaction, is to digest ethyl acetoacetate with ethyl β -bromomethylmalonate in alcoholic solution, when, however, only a poor yield is obtained.

When the sodium derivative of ethyl β -methoxymethylmalonate is treated with methyl iodide, it is converted into ethyl pentane- $\beta\beta\delta\delta$ -tetracarboxylate, $(\text{CO}_2\text{Et})_2\text{CMe}\cdot\text{CH}_2\cdot\text{CMe}(\text{CO}_2\text{Et})_2$. This rearrangement probably takes place in the following way: One molecule of ethyl β -methoxymethylmalonate is methylated in the usual manner with the formation of ethyl β -methoxydimethylmalonate:

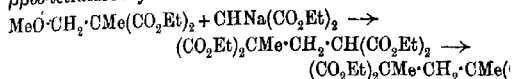


the second molecule is decomposed into formaldehyde and ethyl malonate:

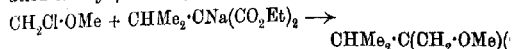


the latter condenses with the ethyl β -methoxydimethylmalonate,

being then further methylated with the formation of ethyl β - β -tetracarboxylate:



The condensation of monochloromethyl ether with ethyl isodimalonate takes place quite normally, and results in the formation of *ethyl β -methoxymethyl- β -isopropylmalonate*,



This ester, on hydrolysis, yields *β -methoxymethyl- β -isopropylmalonic acid*, melting at $83-84^\circ$, which, on heating, loses carbon dioxide to form *α -methoxymethylisovaleric acid*. Further experiments on these substances are in progress.

EXPERIMENTAL.

Condensation of Monochloromethyl Ether and Ethyl Isodimalonate

Finely-divided sodium (11.5 grams) was suspended in diethyl ether (300 c.c.), and ethyl malonate (80 grams) was added. When all had ceased (in about three hours), monochloromethyl ether (50 grams) dissolved in dry ether (50 c.c.), was slowly added to the well-stirred sodium derivative with constant shaking. When no further temperature took place, the mixture, in which much sodium had separated, was allowed to remain for two hours at the same temperature. Water was then added, and the ethereal solution separated was dried and evaporated, when a mobile oil with a faint odour of formaldehyde remained. The oil was rapidly fractionated under diminished pressure (15 mm.), when the following fractions were obtained.

I, up to 115° ; II, $115-140^\circ$; III, $140-220^\circ$ (see p. 1779). Fraction I, which consisted mainly of unchanged ethyl malonate, was refractionated several times until it boiled quite constantly, the boiling fraction in each case being added to fraction II.

Fraction II was then refractionated, when it was found to boil at $121-122^\circ/15$ mm. (yield 50 grams):

0.1820 gave 0.3504 CO_2 and 0.1276 H_2O . $\text{C} = 52.5$; $\text{H} = 7.7$.
 $\text{C}_9\text{H}_{16}\text{O}_6$ requires $\text{C} = 52.9$; $\text{H} = 7.8$ per cent.

Ethyl β -methoxymethylmalonate is a colourless, mobile oil with an odour resembling that of ethyl malonate.

Hydrolysis of Ethyl β-Methoxymethylmalonate.

h *Hydrochloric Acid.*—Ethyl β-methoxymethylmalonate (10 g) was mixed with 50 per cent. hydrochloric acid (50 c.c.) and refluxed for eight hours in a reflux apparatus on the sand-bath. Carbon dioxide was evolved, and, on removing the condenser, the characteristic ethyl acrylate could be distinctly detected.*

The hydrochloric acid solution was evaporated, when a viscous oil remained, which almost completely solidified. After draining on porous porcelain, the acid was crystallised from ether :

i gave 0.2712 CO₂ and 0.0761 H₂O. C = 49.8 ; H = 5.7.

C₈H₉O₄ requires C = 50.0 ; H = 5.6 per cent.

l *Butane-1:3-dicarboxylic acid*, obtained by this method, melted at 131°, whilst Haworth and Perkin (*loc. cit.*) found 135—136° for pure acid. When mixed with a sample of this *cis*-acid which was prepared by Haworth and Perkin, the melting point was found to be unaltered.

l *Over salt* separates as a caseous, white precipitate when silver nitrate is added to a faintly alkaline solution of the ammonium salt. After drying in a vacuum over sulphuric acid, it was analysed :

3 gave 0.0737 Ag. Ag = 60.2.

C₆H₆O₄Ag₂ requires Ag = 60.3 per cent.

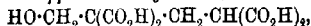
l *Order* to be quite sure that this acid was *cis-cyclobutane-1:3-dicarboxylic acid*, it was digested for half an hour with excess of acetyl chloride, and the solution allowed to evaporate slowly over potassium hydroxide. The anhydride separated as a mass of colourless needles, after draining on porous porcelain, were found to melt at 50°, whilst Haworth and Perkin (*loc. cit.*, p. 338) found the melting point to be 50—51°.

With Barium Hydroxide.—Ethyl β-methoxymethylmalonate (10 g) was boiled with an excess of barium hydroxide solution for 1 hour, when much barium salt had separated and the oil had almost completely dissolved. The solution was cooled, acidified, saturated with common salt, and extracted five times with pure ether. On removal of the ether, an oil was obtained which rapidly solidified. Freeing from traces of oily impurity on porous porcelain, it was crystallised from a mixture of ether and light petroleum :

20 gave 0.1706 CO₂ and 0.0416 H₂O. C = 38.1 ; H = 3.8.

C₈H₁₀O₄ requires C = 38.4 ; H = 4.0.

dicarboxybutane-ααγγ-tetracarboxylic acid,



Experiments are in progress with a view to determining whether this valuable acid can be obtained in quantity by this method.

* XCIII.

crystallises in irregular plates, which melt at 181° with a v evolution of gas. It is readily soluble in water or ethyl acet only sparingly so in ether, light petroleum, or benzene.

When titrated with $N/10$ sodium hydroxide, 0.0618 ne 0.0432 NaOH, whereas a tetrabasic acid, $C_8H_{10}O_8$, should ne 0.0395 NaOH.

When this acid is heated with concentrated hydrochloric a the resulting viscid oil distilled, it is converted into *cis-cyclo* 1:3-dicarboxylic acid.

δ -Hydroxybutane- $\alpha\alpha\gamma$ -tetracarboxylic acid (4 grams) was with concentrated hydrochloric acid (15 c.c.) for half an hou excess of hydrochloric acid was then evaporated, and the residua brown oil purified by distillation under diminished pressure (3 when practically all passed over at $220-230^{\circ}$.

The distillate rapidly solidified when cooled in a freezing n and, after draining on porous porcelain, it was crystallised from 0.1099 gave 0.2002 CO_2 and 0.0591 H_2O . $C=49.7$; $H=5.4$ $C_8H_8O_4$ requires $C=50.0$; $H=5.6$ per cent.

That this acid was *cis-cyclobutane*-1:3-dicarboxylic acid was by its melting point, $127-130^{\circ}$, which was unchanged when with a sample obtained by a different method.

III. With Potassium Hydroxide.—Ethyl β -methoxymethylm (30 grams) was mixed with cold alcoholic potash (30 gr KOH) and allowed to stand overnight, when a large amo a gelatinous potassium salt had separated. Water was add the excess of potassium hydroxide removed by carbon dioxide.

After extracting the unchanged ester with ether, the a solution was freed from alcohol by evaporation, acidified, and ext ten times with ether, when, on evaporation, 13 grams of a v were obtained.

As this acid gave no satisfactory results on analysis, it was with excess of barium carbonate, filtered, and the aqueous s evaporated, when the barium salt, which was more soluble in col in hot water, separated in crystalline crusts. These, when seen the microscope, were found to consist of thin, irregular plates. drying at 100° , the salt was analysed:

0.2026 gave 0.1515 $BaSO_4$. $Ba=44.0$.

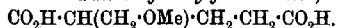
$C_7H_{10}O_5Ba$ requires $Ba=44.1$ per cent.

The pure barium salt was dissolved in water, acidified with hydrochloric acid, and the solution extracted several times wid ether, when a viscid oil was obtained which showed no signs of c lising. After standing for some days in a vacuum over sul acid, it was analysed:

7 gave 0.2286 CO_2 and 0.0793 H_2O . C = 48.9; H = 6.9.

$\text{C}_7\text{H}_{12}\text{O}_5$ requires C = 47.7; H = 6.8 per cent.

ough the analysis was not very satisfactory, it seemed probable
s oil consisted of *α-methoxymethylglutaric acid*,



tempts to convert it into *cyclobutane-1:3-dicarboxylic acid* were
ssful.

Ethyl β-Bromomethylmalonate, $\text{CH}_2\text{Br} \cdot \text{CH}(\text{CO}_2\text{Et})_2$.

he preparation of this ester, ethyl β-methoxymethylmalonate
ms) was mixed with aqueous hydrogen bromide (saturated
100 grams) and allowed to stand for three hours at the ordinary
ture. On pouring into water, a heavy oil separated, which
solved in ether; on evaporation of the washed and dried
l solution, a pale yellow oil was obtained, which was fraction-
der diminished pressure (20 mm.), when practically the whole
l at 120—124°.

ite of its constant boiling point, this oil only gave approximate
on analysis, owing to the ease with which it loses hydrogen
3:

18 gave 0.1392 AgBr. Br = 20.9.

mple which was not distilled gave a slightly more satisfactory

18 gave 0.1579 AgBr. Br = 25.8.

$\text{C}_8\text{H}_{13}\text{O}_4\text{Br}$ requires Br = 31.6 per cent.

l β-bromomethylmalonate is a pale yellow oil with an odour
g that of chloroform.

ction of *Ethyl β-Bromomethylmalonate*.—The ester (5 grams)
solved in dilute acetic acid and gradually treated with zinc dust
ms), the reaction being completed by warming on the water-
The filtered solution was extracted with ether; the ethereal
a washed, dried, and evaporated, and the residual oil was
ysed with excess of alcoholic potash. After removing the
by evaporation, the solution was acidified and extracted with
when a crystalline acid was obtained, which, after recrystallisa-
m ether, melted at 130°:

53 gave 0.1726 CO_2 and 0.0544 H_2O . C = 40.5; H = 5.2.

$\text{C}_4\text{H}_6\text{O}_4$ requires C = 40.7; H = 5.1 per cent.

acid consisted therefore of methylmalonic acid, a conclusion
was confirmed by direct comparison with a sample of methyl-
e acid obtained by a different process.

Ethyl Dicarboxyglutarate, $(\text{CO}_2\text{Et})_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{Et})_2$

(I) It has already been mentioned (p. 1780) in describing the fractionation of the condensation product of monochloromethyl and ethyl sodiomalonate, that a third fraction, boiling at 140° , was obtained. This was carefully refractionated, when it was found to boil constantly at $208^\circ/19$ mm.:

0.1711 gave 0.3361 CO_2 and 0.1148 H_2O . $\text{C}=53.6$; $\text{H}=7.4$
 $\text{C}_{15}\text{H}_{24}\text{O}_8$ requires $\text{C}=54.2$; $\text{H}=7.2$ per cent.

That this substance was undoubtedly ethyl dicarboxyglutarate is shown by the fact that, when hydrolysed with hydrochloric acid, yielded glutaric acid, melting at 97° , and no alteration in the melting point was noticed when it was mixed with a sample of glutaric acid obtained in another way.

(II) The ease with which ethyl β -methoxymethylmalonate comes to solution with loss of methyl alcohol has already been referred to in the preceding section (p. 1779), and is clearly shown by the following experiment.

The low boiling fraction from several condensations, which consisted mainly of ethyl malonate, but which also contained some β -methoxymethylmalonate, was fractionated under the ordinary pressure. After some ethyl malonate had passed over, the thermometer began to rise rapidly, and the residue was therefore distilled under diminished pressure (20 mm.), when it was found to boil at 190 — 210° .

That this was ethyl dicarboxyglutarate was proved by its melting point, and by the fact that, on hydrolysis, it gave glutaric acid.

*Ethyl δ -Methoxybutane- $\alpha\alpha\gamma\gamma$ -tetracarboxylate,
 $\text{MeO}\cdot\text{CH}_2\cdot\text{C}(\text{CO}_2\text{Et})_2\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{Et})_2$*

Ethyl β -methoxymethylmalonate (10 grams) was mixed with anhydride (12 grams) and zinc chloride ($\frac{1}{2}$ gram) and boiled for 10 minutes. The colourless liquid was cooled, poured into water, and the oil which separated was dissolved in ether. After evaporation of the solvent, the residual oil was fractionated under diminished pressure (20 mm.), when, after several fractionations, it distilled at 210 — 215° .

0.1210 gave 0.2417 CO_2 and 0.0797 H_2O . $\text{C}=54.5$; $\text{H}=7.3$
 $\text{C}_{17}\text{H}_{28}\text{O}_8$ requires $\text{C}=54.2$; $\text{H}=7.4$ per cent.

Ethyl δ -methoxybutane- $\alpha\alpha\gamma\gamma$ -tetracarboxylate is a viscid, colourless liquid, possessing practically no odour.

Hydrolysis with Potassium Hydroxide.—The ester (4 grams) was mixed with alcoholic potash (5 grams KOH) and boiled for 10 minutes. The alkaline solution was freed from alcohol, acidified

ed five times with ether, when a viscid oil was obtained which crystallise even on long standing.

Sharp analytical results could not be obtained, an aqueous solution of the acid was made faintly alkaline with ammonia, and the silver salt obtained with silver nitrate. After drying over sulphuric acid, it was analysed :

18 gave 0.1311 CO_2 and 0.0331 H_2O . $\text{C} = 15.8$; $\text{H} = 1.6$.

$\text{C}_9\text{H}_8\text{O}_5\text{Ag}$ requires $\text{C} = 15.6$; $\text{H} = 1.2$ per cent.

It therefore seems little doubt that the oily acid consisted of *cybutane- α,γ,γ -tetracarboxylic acid*.

Hydrolysis with Hydrochloric acid.—Ethyl δ -methoxybutane- α,γ,γ -tricarboxylate (4 grams) was mixed with 50 per cent. hydrochloric acid (5 c.c.) and digested on the sand-bath for twelve hours, when the hydrolysis had taken place. On removing the excess of hydrochloric acid by evaporation, an oil was obtained which rapidly solidified. After draining on porous porcelain, the acid was crystallised, when it melted at $127\text{--}130^\circ$ and consisted of *cis-cyclo-1:3-dicarboxylic acid*.

12 gave 0.2084 CO_2 and 0.0588 H_2O . $\text{C} = 49.8$; $\text{H} = 5.7$.

$\text{C}_6\text{H}_8\text{O}_4$ requires $\text{C} = 50.0$; $\text{H} = 5.6$ per cent.

Methylation of Ethyl β -Methoxymethylmalonate.

As already been mentioned (p. 1779), that when ethyl α -methoxymethylmalonate is treated with sodium ethoxide and methyl iodide the reaction does not take place normally, but results in the formation of ethyl pentane- β,β,β,β -tetracarboxylate.

10 (1.1 grams) was dissolved in alcohol, and to the cold sodium ethoxide, ethyl β -methoxymethylmalonate (10 grams) and methyl iodide (7 grams) were added. The solution, which became slightly turbid, was allowed to stand for two hours, and then heated on the water-bath for fifteen minutes, when it was found to be quite neutral. More sodium ethoxide was added, and the oil which separated was dissolved in ether; the ethereal solution was dried and evaporated, when a colourless oil was obtained which, after two fractionations under diminished pressure (1 mm.), distilled at 215° ; yield 50 per cent. :

18 gave 0.2310 CO_2 and 0.0760 H_2O . $\text{C} = 56.3$; $\text{H} = 7.5$.

$\text{C}_{17}\text{H}_{28}\text{O}_8$ requires $\text{C} = 56.7$; $\text{H} = 7.8$ per cent.

Ethyl pentane- β,β,β,β -tetracarboxylate (3 grams) obtained in this way was mixed with excess of concentrated alcoholic potash and heated for fifteen minutes on the water-bath, when it was completely dissolved. After removing the alcohol by evaporation, the aqueous solution was acidified and extracted with ether, and the ethereal solution was dried and evaporated; a crystalline acid was obtained which, after

recrystallisation from a mixture of ether and benzene, melted with vigorous decomposition.

This acid was shown to consist of pentane- $\beta\beta\delta\delta$ -tetracarboxy by direct comparison with a sample of this acid obtained method described by Guthzeit and Dressel (*Annalen*, 1890, 182).

In order to leave no doubt as to the identity of this acid, it was heated to 180° until no further evolution of carbon dioxide place; the acid solidified on cooling, and was crystallised from

0.1098 gave 0.2115 CO_2 and 0.0734 H_2O . $\text{C} = 52.5$; $\text{H} = 7$
 $\text{C}_7\text{H}_{12}\text{O}_4$ requires $\text{C} = 52.5$; $\text{H} = 7.5$ per cent.

This acid, which melted at $94-95^\circ$, therefore consisted of the *cis*- and *trans*-forms of *s*-dimethylglutaric acid; Guth Dressel (*loc. cit.*) give 91° as the melting point.

When heated with acetyl chloride, it was converted into anhydride, which, after crystallising from light petroleum, m. $93-94^\circ$ (Auwers and K bner, *Ber.*, 1891, 24, 1930, give 95°).

0.1475 gave 0.3183 CO_2 and 0.0930 H_2O . $\text{C} = 58.8$; $\text{H} = 9.1$
 $\text{C}_7\text{H}_{10}\text{O}_3$ requires $\text{C} = 59.1$; $\text{H} = 7.0$ per cent.

Ethyl Carboxy- α -acetylglutarate, $(\text{CO}_2\text{Et})_2\text{CH}\cdot\text{CH}_2\cdot\text{CHAc}\cdot\text{CO}_2\text{Et}$

In preparing this ester, two methods have been used, as stated in the introduction; the following one gives much the more satisfactory result.

Ethyl β -methoxymethylmalonate (40 grams) was mixed with acetoacetate (30 grams), acetic anhydride (40 grams), and zinc chloride (2 grams), and the whole boiled in a reflux apparatus for one hour. The zinc chloride rapidly dissolved, and the mixture became deep red in colour.

After cooling, the solution was poured into water, and the oil separated dissolved in ether; the ethereal solution was washed with water and evaporated, and the residual oil was fractionated under diminished pressure (20 mm.). After some unchanged ethyl β -methoxymethylmalonate had passed over, the temperature rapidly rose, and the remainder distilled constantly at 197° . The yield was 10 per cent.:

0.0865 gave 0.1751 CO_2 and 0.0588 H_2O . $\text{C} = 55.2$; $\text{H} = 7.5$
 $\text{C}_{14}\text{H}_{22}\text{O}_7$ requires $\text{C} = 55.6$; $\text{H} = 7.3$ per cent.

Ethyl carboxy- α -acetylglutarate is a pale yellow oil with a faint ethereal odour. Its alcoholic solution gives an intense purplish coloration with ferric chloride.

Hydrolysis of Ethyl Carboxy- α -acetylglutarate.—The ester (3 g) was mixed with concentrated hydrochloric acid (25 c.c.) and heated

apparatus for six hours, the condenser being removed from time to time to allow the alcohol to escape. On evaporating the hydrochloric acid, a viscid oil was obtained, slowly solidified and evidently consisted of the hydrate of butyric acid. The semicarbazone prepared from this melted at 176° (Haworth and Perkin, Trans., 1908, 93, 589), and this point was unaltered when the substance was mixed with a portion of the semicarbazone of γ -acetobutyric acid obtained by a different method.

*Condensation of Monochloromethyl Ether and Ethyl
isopropylsodiummalonate.*

In carrying out this condensation, finely-divided sodium (11.5 grams) suspended in anhydrous ether (300 c.c.), and ethyl isopropylmalonate (101 grams) was added. A vigorous reaction immediately took place, and in about two hours all the sodium had dissolved, no new compound, however, separating out.

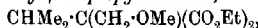
The solution was cooled, and monochloromethyl ether (50 grams) added in twice its volume of dry ether was gradually added, care being taken that no rise in temperature should take place. Much sodium chloride immediately separated, and, after standing for two days at the ordinary temperature, the mixture was heated on the water-bath for fifteen minutes. Water was then added, and the ether separated, dried, and evaporated.

The residual oil was fractionated under diminished pressure (20 mm.), and, after several fractionations, the main portion boiled at 132° :

435 gave 0.3102 CO_2 and 0.1137 H_2O . $\text{C} = 58.9$; $\text{H} = 8.9$.

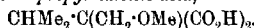
$\text{C}_{12}\text{H}_{22}\text{O}_5$ requires $\text{C} = 58.5$; $\text{H} = 8.9$ per cent.

Ethyl β -methoxymethyl- β -isopropylmalonate,



colourless, mobile oil possessing a pleasant ethereal odour.

Methoxymethyl- β -isopropylmalonic acid,



The pure ester (40 grams) was mixed with excess of alcoholic sodium hydroxide (30 grams KOH) and allowed to stand in the dark overnight. It was then boiled until hydrolysis was complete; water was added, and the solution evaporated until free from alcohol, then dried, saturated with ammonium sulphate, and extracted ten times with ether. The ethereal solution was dried and evaporated, when a solid oil was obtained. In order to obtain the pure acid, the oil was dissolved in water and boiled with an excess of barium carbonate; on filtering the filtrate, the barium salt separated in crystalline

crusts, which, under the microscope, were seen to consist of microscopic plates. After drying at 100° , it was analysed :

0.1300 gave 0.0903 BaSO_4 . Ba = 40.9.

0.1425 „ 0.0990 BaSO_4 . Ba = 40.9.

$\text{C}_8\text{H}_{12}\text{O}_5\text{Ba} \cdot \frac{1}{2}\text{H}_2\text{O}$ requires Ba = 41.1 per cent.

The barium salt was dissolved in water, the solution acidified, and extracted ten times with washed ether, when a colourless oil was obtained which gradually solidified. After draining on porous porcelain, it was crystallised from a mixture of benzene and light petroleum, from which it separated in colourless plates :

0.1450 gave 0.2671 CO_2 and 0.0943 H_2O . C = 50.2; H = 7.2.

$\text{C}_8\text{H}_{14}\text{O}_5$ requires C = 50.5; H = 7.4.

β -Methoxymethyl- β -isopropylmalonic acid melts at 83 – 84° . It is readily soluble in water, benzene, ether, or chloroform, but only sparingly so in light petroleum, even on boiling.

β -Methoxymethylisovaleric Acid, $\text{CHMe}_2\cdot\text{CH}(\text{CH}_2\cdot\text{OMe})\cdot\text{CO}_2\text{H}$.

β -Methoxymethyl- β -isopropylmalonic acid (30 grams) was heated at 180° for half an hour, when all evolution of carbon dioxide had ceased.

The crude monobasic acid was mixed with alcohol (50 c.c.) and sulphuric acid (5 c.c.), and heated in a reflux apparatus for four hours on the water-bath. The alcoholic solution was poured into ether, and the ethereal solution washed, dried, and evaporated. The residual oil was fractionated under diminished pressure (100 mm.), when practically the whole distilled at 120 – 130° . After two fractionations, it boiled constantly at $126^{\circ}/100$ mm. :

0.1179 gave 0.2699 CO_2 and 0.1047 H_2O . C = 62.5; H = 9.9.

$\text{C}_9\text{H}_{18}\text{O}_3$ requires C = 62.1; H = 10.3 per cent.

Ethyl β -methoxymethylisovalerate is a colourless, mobile oil with a pungent odour resembling that of ethyl isovalerate. In order to obtain the acid in a pure state, the ester (19 grams) was mixed with concentrated alcoholic potassium hydroxide (10 grams KOH) and allowed to stand in the cold overnight, when it was found that nearly complete hydrolysis had taken place. Water was added, and the excess of alkali removed with carbon dioxide.

After extracting any unchanged ester with ether, the solution was freed from alcohol by evaporation, acidified, and the acid extracted several times with ether. The ether was dried and evaporated, and the residual oil fractionated under diminished pressure (100 mm.), when it was found to boil fairly constantly at 165 – 170° .

Sharp analytical results could not be obtained, owing to the tendency of the acid to lose methyl alcohol, as is shown by the following

methoxyl determinations, which were made with two different preparations:

0.3761 gave 0.5885 AgI. MeO = 20.6.

0.1903 „ 0.2619 AgI. MeO = 18.1.

$C_7H_{14}O_2$ requires MeO = 21.2 per cent.

β-Methoxymethylisovaleric acid is a colourless oil possessing a very rancid odour. It is readily soluble in most organic solvents. Owing to the difficulty experienced in obtaining the acid in a pure state, the silver salt was prepared by adding silver nitrate to a faintly alkaline solution of the ammonium salt; it is readily soluble in hot water, and separates, on cooling, in colourless needles:

0.1055 gave 0.1274 CO_2 and 0.0461 H_2O . C = 32.9; H = 5.0.

0.1162 „ 0.0501 Ag. Ag = 43.1.

$C_7H_{13}O_3Ag$ requires C = 33.2; H = 5.1; Ag = 42.7 per cent.

The author wishes to thank Professor W. H. Perkin for the interest which he has taken in this work, and also Mr. F. B. Dehn, M.Sc., for carrying out some of the preliminary experiments and some of the analyses. Much of the expense of this research has been met by a grant from the Research Fund Committee of the Chemical Society, for which the author is much indebted.

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CLXXVI.—*The Effect of Constitution on the Rotatory Power of Optically Active Nitrogen Compounds.*
Part III.

By REGINALD WILLIAM EVERATT and HUMPHREY OWEN JONES.

IN continuing the investigation of the relation of optical rotatory power to constitution in the optically active quinquivalent nitrogen compounds (Thomas and Jones, *Trans.*, 1906, **89**, 280; Jones and Hill, *Trans.*, 1908, **93**, 295; Everatt, *ibid.*, 1225), it appeared to be of interest to examine compounds containing the *p*-tolyl group in order to compare them with those containing the *p*-bromophenyl group which had been examined previously.

Three compounds having the requisite structure and containing the *p*-tolyl group have been prepared and examined by Wedekind (*Zeitsch. physikal. Chem.*, 1903, **45**, 235) and Wedekind and Oberheide (*Ber.*, 1904, **37**, 2712), but attempts to resolve these compounds into

their optically active constituents by means of camphorsulphonic acid were unsuccessful.

So far, we have examined only one of these compounds, namely, *p*-tolylbenzylmethylallylammonium iodide, and this has been resolved into its optically active constituents by crystallising its hydrogen tartrate from alcohol and ether. We have made determinations of the rotatory power of the active ion and of the active iodide. The results are described in this paper, and compared with those already obtained for the corresponding compounds containing the phenyl or the *p*-bromophenyl group.

This is another instance to be added to those already described, in which resolution can be effected by means of the weak acids, tartaric or camphoric acid, when the much stronger acids, camphorsulphonic or bromocamphorsulphonic acid, have failed to effect resolution.

p-Tolylbenzylmethylallylammonium iodide was prepared by the union of benzylmethyl-*p*-toluidine and allyl iodide mixed in molecular proportions; the mixture slowly darkened and deposited a crystalline solid, but we never observed the violent combination described by Wedekind and Oberheide (*loc. cit.*). When dried on a porous plate and crystallised twice from alcohol and ether, the salt was obtained as a white, crystalline powder, which melted sharply at 145–146°.

p-Tolylbenzylmethylallylammonium hydrogen d-tartrate was prepared by mixing molecular proportions of the ammonium hydroxide and tartaric acid in dilute alcoholic solution and evaporating on a water-bath until the solution became turbid; alcohol was now added until the solution was clear, and from this the salt was precipitated by the addition of ether as a fine, crystalline powder with a slight brown colour. The addition of ether was found to require great care, as its too rapid addition resulted in the deposition of an oil, which, if deposited in any considerable quantity, prevented the crystallisation of the salt. When the oil was deposited in small quantity only, then, on standing, the sticky precipitate gradually became crystalline. The salt was then recrystallised in the same way, using the same precautions, and was thus obtained in clusters of white needles melting at 84–85°. The rotatory power of the salt in aqueous solution was found to diminish, to become negative, and gradually to approach a constant value after about thirteen recrystallisations:

0.2133 gave 0.4884 CO₂ and 0.1395 H₂O. C = 62.7; H = 7.3.

C₂₂H₂₇O₆N, H₂O requires C = 62.95; H = 6.95 per cent.

This salt, like the other hydrogen tartrates of substituted ammonium ions, crystallises with water of crystallisation even when deposited from absolute alcohol.

The following determinations of rotatory power were made, a 2-dm. tube being used in all cases:

After thirteen recrystallisations, 0.1248 gram in 20.059 grams of solution gave $[\alpha]_D -0.60^\circ$ at 20° ; hence $[\alpha]_D -48.2^\circ$ and $[M]_D -202^\circ$; 0.1041 gram in 20.436 grams of solution gave $\alpha_D -0.49^\circ$; hence $[\alpha]_D -48.1^\circ$ and $[M]_D -201^\circ$.

After fifteen recrystallisations, 0.1006 gram in 17.930 grams of solution gave $\alpha_D 0.54^\circ$; hence $[\alpha]_D -48.1^\circ$ and $[M]_D -201^\circ$.

Taking the value of $[M]_D$ for hydrogen tartrate ion to be $+42^\circ$ at 20° (Landolt), the value of $[M]_D$ for the basic ion is -243° at 20° .

The influence of temperature on the rotatory power of this salt was investigated in the usual way, and the values of the molecular rotatory powers of the ion for the temperatures between 0° and 50° were deduced. The densities of the solutions have been taken to be those of water at the same temperatures, and Landolt's values for the rotatory powers of the hydrogen tartrate ion have been taken.

0.1144 Gram of salt in 20.815 grams of solution :

<i>t</i> .	α_D .	$[\alpha]_D$.	$[M]_D$.	$[M]_D$ for basic ion.
5°	-0.55°	-50.0°	-310°	-252°
12	0.54	49.5	207	249
22	0.53	48.1	202	244
34	0.52	47.1	197	239
40	0.505	45.6	191	233
51	0.50	44.93	188	230

The value of $[M]_D$ for the basic ion at 15° may therefore be taken as -246° .

p-Tolylbenzylmethylallylammonium iodide was precipitated from aqueous solutions of the tartrate by neutralising with sodium carbonate and adding excess of concentrated potassium iodide solution. The active iodide was thus obtained in large, lustrous needles, melting at $144-145^\circ$. A mixture of the active and inactive iodides melted at $143-144^\circ$.

Determinations of the rotatory power of the levorotatory iodide were made in alcohol and in chloroform solution :

In alcohol: 0.0676 gram in 15.030 grams of solution ($d=0.800$) gave $\alpha_D -0.48^\circ$; hence $[\alpha]_D -66.7^\circ$ and $[M]_D -258^\circ$. 0.0752 Gram in 13.482 grams of solution ($d=0.808$) gave $\alpha_D -0.60$; hence $[\alpha]_D -66.5^\circ$ and $[M]_D -257.4^\circ$.

In chloroform: 0.085 gram in 27.360 grams of solution ($d=1.491$) gave $\alpha_D -0.62^\circ$ fifteen minutes after making up the solution; hence $[\alpha]_D -66.9^\circ$ and $[M]_D -259^\circ$.

After one hour, $\alpha_D -0.57^\circ$; after two hours, $\alpha_D -0.53^\circ$; after four hours, $\alpha_D -0.47^\circ$; after eight hours, $\alpha_D -0.41^\circ$, and after thirty-six hours the solution was inactive; hence the value for $[M]_D$ at the time of making up the solution is -267.4° .

A second determination with 0.0639 gram in 25.218 grams of

solution ($d = 1.490$) gave $\alpha_D -0.52^\circ$; hence $[\alpha]_D -66.8^\circ$ and $[M]_D -261^\circ$.

A comparison of these results with those obtained for the corresponding phenyl compound (Pope and Harvey, *Trans.*, 1901, 79, 828) and bromophenyl compound (Everatt, *Trans.*, 1908, 93, 1236) is made in the following table:

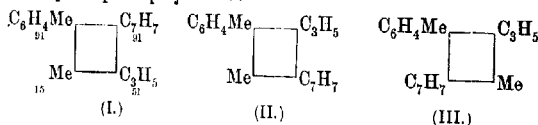
	Melting point of iodide.	$[M]_D$ for ion at 15° .	$[M]_D$ for iodide.	
			in alcohol.	in chloroform.
Benzylmethylallyl.				
Phenyl	140–142°	167°	—	201°
<i>p</i> -Bromophenyl	134–135	191	–242°	–346
<i>p</i> -Tolyl	145–146	–246	–257	–267

It is noteworthy that the rotatory power of the ion of the compound containing the *p*-tolyl group is much greater than that of either of the corresponding compounds containing the phenyl or the *p*-bromophenyl group, although the latter is much the heaviest of the three groups. Further, although in the compound now described there are two groups, the benzyl and *p*-tolyl groups, which have identical masses, the rotatory power is still large. This may well be discussed in connexion with the values for the product of asymmetry for this compound.

The difference between the observed values for the rotatory power of the iodide in alcohol and in chloroform is unusually small in this case, and reminds one of the case of phenylbenzylmethylisobutylammonium iodide (Thomas and Jones, *loc. cit.*).

A consideration of the values of the product of asymmetry, calculated for this compound according to the method used in former communications, shows at once that it is not possible to reconcile the experimental results with these values.

Taking the three possible configurations for the compound represented by the plane projections:



we obtain the values for the product of asymmetry which are given in the table, together with those for the corresponding phenyl and *p*-bromophenyl compounds.

	$[M]_D$ for ion at 15° .	$P_1 \times 10^2$.	$P_2 \times 10^2$.	$P_3 \times 10^2$.
Phenyl	167°	0.07	4.87	–1.66
<i>p</i> -Bromophenyl	191	–7.7	0.71	14.6
<i>p</i> -Tolyl	246	3.9	0.00	3.9

No assumptions as to the configuration of these three compounds will give values of p which are in the same order as the values of the

molecular rotatory powers, and we are therefore forced to the conclusion that the constitution of the groups exerts so great an influence on the magnitude of the rotatory power that the effect of mass is largely veiled by it.

Other compounds containing the *p*-tolyl group are now under investigation, and it is hoped that two complete series, corresponding with the two series of compounds containing the phenyl group, will be resolved, so that we can again compare the effects produced by the different aliphatic groups on the rotatory powers of the ions containing them.

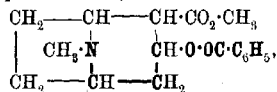
The expenses of this investigation were defrayed by grants made by the Government Grant Committee of the Royal Society, for which we are glad to make this grateful acknowledgment.

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CLXXVII.—*Relation between Chemical Constitution and Physiological Action in Certain Substituted Aminoalkyl Esters.*

By FRANK LEE PYMAN.

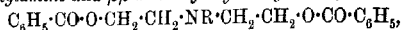
THE desirability of producing a local anæsthetic to take the place of cocaine has led to the preparation of substances designed to possess the characteristic action of this alkaloid, whilst at the same time being free from its toxic effects. In recent years, investigation has chiefly been directed to substituted aminoalkyl esters, which contain a skeleton, $\text{:N}\cdot\dot{\text{C}}\cdot\dot{\text{C}}\cdot\text{O}\cdot\text{CO}\cdot\text{R}$, similar to that part of the cocaine molecule which is printed heavily in the formula :



and certain substances of this type, for example, "stovaine," "alypin," and "novocaine," have found application in medicine as local anæsthetics. The present paper records the preparation and the chemical, physical, and physiological properties of certain new aminoalkyl esters. Several of these possess local anæsthetic properties, some in a pronounced degree, but none are suitable for use in medicine as local anæsthetics, owing in some cases, to irritation produced on injec-

tion, in others to a relatively high general toxicity. The physiological examination of these substances (as hydrochlorides, except where otherwise stated) was carried out by Messrs. H. H. Dale and C. T. Symons, of the Wellcome Physiological Research Laboratories, to whom the author wishes to tender his best thanks.

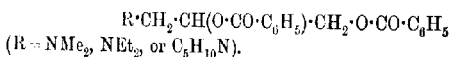
The nitrogen atom in cocaine is connected directly to one alkyl (methyl) group and indirectly through two chains of methylene (or substituted methylene) groups to a benzoylated secondary alcoholic group. It therefore appeared of interest to prepare $\beta\beta$ -dibenzoyloxy-methyl-diethylamine and $\beta\beta$ -dibenzoyloxytriethylamine,



which contain a nitrogen atom connected directly to one alkyl group and indirectly through two methylene groups to two benzoylated alcohol groups.

These substances were consequently prepared by benzoylating the corresponding amino-alcohols. The methyl derivative has no local anæsthetic action, and the ethyl derivative very little. Marked differences are shown by the two substances in other respects; in the rabbit, the ethyl derivative causes respiratory and cardiac depression, whilst the methyl derivative has no marked action on the heart or respiration, but causes marked extensor convulsions reminiscent of those produced by a small dose of strychnine; in the frog, the methyl derivative causes primarily paralysis of the brain centres, whilst the ethyl derivative causes central paralysis, primarily affecting the spinal cord.

The next point investigated was the introduction of two benzoyl groups into the molecule in a different manner, the substances prepared being respectively $\beta\gamma$ -dibenzoyloxydimethylpropylamine, $\beta\gamma$ -dibenzoyloxydiethylpropylamine, and $\beta\gamma$ -dibenzoyloxy-1-propylpiperidine,

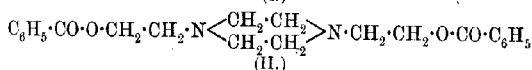
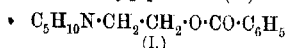


These substances possess a very considerable local anæsthetic action, but their value is discounted by the fact that they are distinctly toxic and irritant, and also acid in reaction. Here, again, the diethyl derivative has a depressant action on the heart, which is not observable in the case of the dimethyl derivative. Both the dimethyl and diethyl derivatives act on the central nervous system of the mammal, producing spasms faintly reminiscent of those caused by strychnine, the dimethyl being the most active in this respect. In the frog, the dimethyl derivative quickly paralyses the brain, but its action on the spinal cord is comparatively slight, whilst the diethyl derivative has a paralytic effect on the brain and spinal cord, the latter being at least as markedly and rapidly affected as the former.

The methobromide of the dimethyl derivative was also tested, and found to have a considerably feebler general toxicity than the hydrochloride, the central convulsant action of the latter on the mammal being replaced or obscured by a curare-like effect in the methobromide.

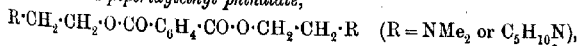
The above benzoates were prepared from the corresponding amino-dihydroxypropanes of C. F. Roth (*Ber.*, 1882, 15, 447). Roth's statements show that he had some difficulty in purifying dihydroxy-piperidylpropane, and he does not state its melting point, but the substance may readily be purified by distillation under 80 mm. pressure, when it boils at 200–201° and solidifies on cooling; subsequent crystallisation from acetone yields the substance in large, monoclinic plates, melting at 79–80°. In the same communication, Roth gives an account of some attempts to prepare the dibenzoates of these amino-alcohols, but he was unable to isolate the dibenzoyloxy-derivatives of diethylpropylamine and 1-propylpiperidine, and only obtained β -dibenzoyloxydimethylpropylamine in the form of its picrate, for which he gives the melting point 112°. The dibenzoyloxy-derivatives of the three bases have now been prepared by the Schotten-Baumann reaction, but β -dibenzoyloxydimethylpropylamine picrate, prepared from the pure crystalline hydrochloride, has been found to melt at 178–179°.

Certain dialkylaminoethyl benzoates described by E. Schering (D.R.P. 175080) have been stated to be useful local anæsthetics, and it therefore seemed of interest to prepare *piperidylethyl benzoate* (I), and *s-di- β -benzoyloxy-1:4-diethylpiperazine* (II):



These bases were obtained by benzoylating the corresponding amino-alcohols, of which one, 1:4-diethanolpiperazine, has not previously been described. Piperidylethyl benzoate shows some local anæsthetic property, but is distinctly irritant; its methobromide is neither anæsthetic nor irritant. *s-Di- β -benzoyloxy-1:4-diethylpiperazine* shows very distinct local anæsthetic properties, but is the most poisonous of the series, 50 milligrams causing rapid death in a rabbit weighing 1 kilogram; the appearance of the blood *post mortem* showed that the hæmoglobin was decomposed.

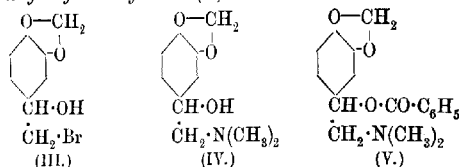
In two cases the phthaloyl group had been introduced into the molecule in the place of the benzoyl group; thus *diethylaminoethyl phthalate* and *piperidylethyl phthalate*,



have been prepared by the interaction of phthaloyl chloride and the corresponding amino-alcohol in ethereal solution.

Diethylaminoethyl phthalate hydrochloride was submitted to physiological examination, and appeared to have neither anæsthetic nor irritant properties.

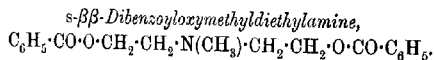
β -Bromo- α -hydroxy-3 : 4-methylenedioxyethylbenzene (Barger and Jowett, Trans., 1905, 87, 970) formed the starting point of another attempt. By condensing this compound (III) with dimethylamine, β -hydroxy- β -3 : 4-methylenedioxyphenylethyldimethylamine (IV) was obtained, and this yielded, on benzylation, β -benzoyloxy- β -3 : 4-methylenedioxyphenylethyldimethylamine (V) :



This substance is a substituted aminoalkyl benzoate, and has very considerable anæsthetic properties, but is unfortunately also toxic and irritant.

EXPERIMENTAL.

Those compounds described below which are aminoalkyl benzoates were prepared by the Schotten-Baumann method. The corresponding hydroxy-derivative was dissolved in a large quantity of 10 per cent. sodium hydroxide solution, and well shaken with a large excess of benzoyl chloride. The crude ester was then extracted with ether, removed from the ether with dilute hydrochloric acid, regenerated with sodium carbonate solution, and again dissolved in ether. After the ether had been dried and distilled, the resulting aminoalkyl benzoate was purified in a suitable manner in each special case.



Seventeen grams of *s*- β -dihydroxymethylethylamine gave, on benzylation, 36 grams of *s*- β -dibenzoyloxymethyldiethylamine, a yield of 77 per cent. of the theoretical. This was purified by crystallisation of the hydrogen oxalate, and regenerated from the pure salt ; it formed a colourless, viscid oil which did not crystallise.

The hydrochloride separates from absolute alcohol in diamond-shaped plates, which melt at 132–133°. It is readily soluble in water, giving an acid solution, and sparingly so in absolute alcohol. This salt is anhydrous ;

0.2474 gave 0.5692 CO_2 and 0.1228 H_2O . $\text{C} = 62.7$; $\text{H} = 6.0$.

0.2584 „ 0.1020 AgCl , $\text{Cl} = 9.8$.

$\text{C}_{19}\text{H}_{21}\text{O}_4\text{N}, \text{HCl}$ requires $\text{C} = 62.7$; $\text{H} = 6.0$; $\text{Cl} = 9.7$ per cent.

The *hydrobromide* was obtained, by evaporation of its aqueous solution, as an oil which gradually solidified, forming clusters of hard plates. After recrystallisation from dilute alcohol, it melts at $124-126^\circ$. It is anhydrous, and is almost insoluble in water, but fairly readily soluble in alcohol:

0.1150 gave 0.0540 AgBr . $\text{Br} = 20.0$.

$\text{C}_{19}\text{H}_{21}\text{O}_4\text{N}, \text{HBr}$ requires $\text{Br} = 19.6$ per cent.

The *hydrogen oxalate* crystallises from boiling water in prismatic needles, melting at $169-170^\circ$; it is anhydrous, and is very sparingly soluble in cold water or alcohol:

0.2030 gave 0.4476 CO_2 and 0.1002 H_2O . $\text{C} = 60.1$; $\text{H} = 5.5$.

$\text{C}_{19}\text{H}_{21}\text{O}_4\text{N}, \text{C}_2\text{H}_2\text{O}_4$ requires $\text{C} = 60.4$; $\text{H} = 5.6$ per cent.

The *aurichloride* separates from alcohol in clusters of hard, prismatic, yellow needles, which melt at $109-110^\circ$; it is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol:

0.1400 gave 0.0411 Au . $\text{Au} = 29.4$.

$\text{C}_{19}\text{H}_{21}\text{O}_4\text{N}, \text{HAuCl}_4$ requires $\text{Au} = 29.6$ per cent.

The *platinichloride* crystallises from alcohol in clusters of fine yellow needles, which begin to sinter at 87° and melt clearly at 95° ; it contains four molecular proportions of water of crystallisation, and is insoluble in water, but sparingly soluble in alcohol:

0.1170, air-dried, gave 0.1694 CO_2 and 0.0502 H_2O . $\text{C} = 39.5$; $\text{H} = 4.8$.

0.0978 „ lost 0.0058 at 100° . $\text{H}_2\text{O} = 5.9$.

0.0920, dried at 100° , gave 0.0167 Pt . $\text{Pt} = 18.2$.

$(\text{C}_{19}\text{H}_{21}\text{O}_4\text{N})_2\text{H}_2\text{PtCl}_6, 4\text{H}_2\text{O}$ requires $\text{C} = 40.1$; $\text{H} = 4.6$; $\text{H}_2\text{O} = 6.3$ per cent.

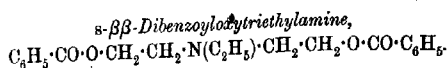
$(\text{C}_{19}\text{H}_{21}\text{O}_4\text{N})_2\text{H}_2\text{PtCl}_6$ requires $\text{Pt} = 18.3$ per cent.

The *picrate* crystallises from alcohol in long, slender, yellow needles, which melt at $136-137^\circ$.

The *methobromide* was obtained in long, fine needles by the addition of methyl bromide to a cold alcoholic solution of the base. After crystallisation from alcohol, it formed long, fine needles, which melted at $170-171^\circ$. It is insoluble in water, but sparingly soluble in cold alcohol:

0.1676 gave 0.0744 AgBr . $\text{Br} = 18.2$.

$\text{C}_{19}\text{H}_{21}\text{O}_4\text{N}, \text{CH}_3\text{Br}$ requires $\text{Br} = 18.9$ per cent.



Ten grams of *s-ββ*-dihydroxytriethylamine gave, on benzoylation, 18 grams of *s-ββ*-dibenzoyloxytriethylamine, the yield being 70 per cent. of the theoretical. This was purified by crystallisation of the hydrogen oxalate, and regenerated from the pure salt; it formed a colourless, viscid oil, which did not crystallise.

The *hydrochloride* separated from its aqueous solution as a viscid oil, which gradually solidified, after the addition of a little alcohol, on evaporation in a vacuum over sulphuric acid. On recrystallisation from benzene, it was obtained in matted clusters of fine needles, which melted at 110—111°, and contained half a molecular proportion of alcohol of crystallisation; the alcohol is gradually lost at 110°, but is not entirely removed before further decomposition of the salt commences. This salt is very readily soluble in water, giving an acid solution, and readily so in alcohol:

0.1410 gave 0.3260 CO₂ and 0.0874 H₂O. C = 63.1; H = 6.9.

0.2582 „ 0.0960 AgCl. Cl = 9.2.

C₂₀H₂₃O₄N, HCl, $\frac{1}{2}$ C₂H₆O requires C = 62.9; H = 6.8; Cl = 8.8 per cent.

The *hydrogen oxalate* crystallises from boiling water in glistening needles, which melt at 157—158°; it is anhydrous, and is very sparingly soluble in cold water or alcohol:

0.2106 gave 0.4734 CO₂ and 0.1096 H₂O. C = 61.3; H = 5.8.

C₂₀H₂₃O₄N, C₂H₂O₄ requires C = 61.2; H = 5.7 per cent.

The *aurichloride* separates from alcohol in hard, yellow nodules of prismatic needles, which melt at 115—116°. It is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol:

0.2940 gave 0.0850 Au. Au = 28.9.

C₂₀H₂₃O₄N, HAuCl₄ requires Au = 28.9 per cent.

The *platinichloride* crystallises from alcohol in fine, yellowish-brown needles, which have no definite melting point, but commence to soften and lose alcohol at 60°, and are completely molten at 95°. It contains one and a-half molecular proportions of alcohol of crystallisation, and is insoluble in water, and very sparingly soluble in alcohol:

0.0978, air-dried, gave 0.1594 CO₂ and 0.0416 H₂O. C = 44.4; H = 4.8.

0.1020 „ lost 0.0064 at 100°. C₂H₆O = 6.3.

0.1354, dried at 100°, gave 0.0244 Pt. Pt = 18.0.

(C₂₀H₂₃O₄N)₂, H₂PtCl₆, $1\frac{1}{2}$ C₂H₆O requires C = 44.4; H = 4.8;
C₂H₆O = 6.0 per cent.

(C₂₀H₂₃O₄N)₂, H₂PtCl₆ requires Pt = 17.8 per cent.

The *picrate* crystallises from alcohol in microscopic rosettes of yellow needles, which melt at 136—137°.

βγ-Dibenzoyloxydimethylpropylamine,
 $(\text{CH}_3)_2\text{N}\cdot\text{CH}_2\cdot\text{CH}(\text{O}\cdot\text{CO}\cdot\text{C}_6\text{H}_5)\cdot\text{CH}_2\cdot\text{O}\cdot\text{CO}\cdot\text{C}_6\text{H}_5,$

Eighteen grams of *βγ*-dihydroxydimethylpropylamine gave, on benzoylation, 37 grams of *βγ*-dibenzoyloxydimethylpropylamine, a yield of 75 per cent. of the theoretical. The substance was purified by crystallisation of the picrate and regeneration from the pure salt, when it formed a colourless, viscid oil which did not crystallise.

The *hydrochloride* crystallises from ethyl acetate in long needles, which melt at 179—180°. It is anhydrous, and is very readily soluble in water, giving an acid solution, but fairly readily so in alcohol :

0.1967 gave 0.4493 CO₂ and 0.1079 H₂O. C = 62.3 ; H = 6.1.

0.2076 „ 0.0794 AgCl. Cl = 9.5.

C₁₉H₂₁O₄N.HCl requires C = 62.7 ; H = 6.1 ; Cl = 9.7 per cent.

The *nitrate* separates from absolute alcohol in large, acicular crystals, which melt at 111—112°. It is anhydrous, and is sparingly soluble in cold water or alcohol :

0.1408 gave 0.3004 CO₂ and 0.0722 H₂O. C = 58.2 ; H = 5.7.

C₁₉H₂₁O₄N.HNO₃ requires C = 58.4 ; H = 5.7 per cent.

The *aurichloride* separates from alcohol in clusters of glistening, golden-yellow needles, which melt at 161—162°. It is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol :

0.1417 gave 0.0416 Au. Au = 29.4.

C₁₉H₂₁O₄N.HAuCl₄ requires Au = 29.6 per cent.

The *platinichloride* crystallises from water in clusters of pale yellow needles, which melt at 174—176°. It is anhydrous, and is insoluble in water, but very sparingly soluble in cold alcohol :

0.1275 gave 0.0230 Pt. Pt = 18.0.

(C₁₉H₂₁O₄N)₂.HPtCl₆ requires Pt = 18.3 per cent.

The *methobromide* crystallises from ethyl acetate in clusters of monoclinic plates, which melt at 94—95°. It contains one molecule of water of crystallisation, and is readily soluble in water or alcohol :

0.1334, air-dried, gave 0.2660 CO₂ and 0.0710 H₂O. C = 54.4 ; H = 6.0.

0.2288 „ 0.0966 AgBr. Br = 18.0.

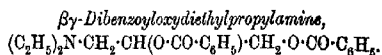
C₁₉H₂₁O₄N.CH₃Br.H₂O requires C = 54.5 ; H = 6.0 ; Br = 18.2 per cent.

The *picrate* has already been described by Roth (*loc. cit.*), and characterised as yellow, silky plates (from water) which melted at 112°. For comparison, it has been prepared from the pure hydrochloride by precipitation with picric acid, and recrystallised from alcohol. It formed glistening, yellow scales, which melted at

178—179°. After recrystallisation from water, the substance melted at the same temperature, and, on analysis, proved to be the normal salt:

0.2004 gave 0.3952 CO₂ and 0.0784 H₂O. C = 53.8; H = 4.4.

C₁₉H₂₁O₄N, C₆H₅O₂N₃ requires C = 53.9; H = 4.4 per cent.



Twenty grams of *β*-dihydroxydiethylpropylamine gave, on benzylation, 40 grams of *β*-dibenzoyloxydiethylpropylamine, a yield of 83 per cent. of the theoretical. The substance was purified by crystallisation of the picrate, and regeneration from the pure salt, when it formed a colourless, viscid oil which did not crystallise.

The *hydrochloride* crystallises from ethyl acetate in colourless, highly-refracting plates, which melt at 125—126°. It is anhydrous, and is very readily soluble in water, giving an acid solution, but sparingly so in cold alcohol or ethyl acetate:

0.2678 gave 0.6334 CO₂ and 0.1572 H₂O. C = 64.5; H = 6.6.

0.2324 „ 0.0854 AgCl. Cl = 9.1.

C₂₁H₂₅O₄N, HCl requires C = 64.3; H = 6.7; Cl = 9.1 per cent.

The *nitrate* crystallises from ethyl acetate in clusters of short needles, which melt at 123—124°. It is anhydrous, and is sparingly soluble in water, but fairly readily so in cold alcohol or ethyl acetate:

0.1502 gave 0.3292 CO₂ and 0.0818 H₂O. C = 59.8; H = 6.1.

C₂₁H₂₅O₄N, HNO₃ requires C = 60.2; H = 6.3 per cent.

The *hydrogen oxalate* separates from boiling water in clusters of imperfectly-formed, acicular crystals, which melt at 120—121°. It is anhydrous, and is sparingly soluble in water, but very sparingly so in alcohol:

0.1346 gave 0.3058 CO₂ and 0.0744 H₂O. C = 62.0; H = 6.2.

C₂₁H₂₅O₄N, C₂H₂O₄ requires C = 62.0; H = 6.1 per cent.

The *aurichloride* crystallises from alcohol in clusters of long, yellow, silky needles, which melt at 121—122°. It is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol:

0.3138 gave 0.0900 Au. Au = 28.7.

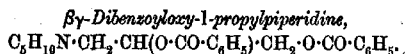
C₂₁H₂₅O₄N, HAuCl₄ requires Au = 28.4 per cent.

The *platinichloride* crystallises from alcohol in hard clusters of yellowish-brown, monoclinic plates, which melt at 176—177°. It is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol:

0.1028 gave 0.0176 Pt. Pt = 17.1.

(C₂₁H₂₅O₄N)₂·H₂PtCl₆ requires Pt = 17.4 per cent.

The *picrate* crystallises from dilute alcohol in radial clusters of yellow needles, which melt at 139—140°.



Twenty grams of β -dihydroxy-1-propylpiperidine gave, on benzoylation, 40 grams of β -dibenzoyloxy-1-propylpiperidine in the form of a colourless, viscid oil; this solidified on stirring with a little alcohol, and, after recrystallisation from the same solvent, yielded 30 grams of pure base, that is, 65 per cent. of the theoretical.

The base crystallises from alcohol in clusters of long, white, glistening needles, which melt at 64—65°; it is insoluble in water, and somewhat sparingly soluble in cold alcohol:

0.1630 gave 0.4320 CO_2 and 0.1010 H_2O . C = 72.3; H = 6.9.

$\text{C}_{22}\text{H}_{25}\text{O}_4\text{N}$ requires C = 71.9; H = 6.9 per cent.

The *hydrochloride* crystallises from benzene in clusters of monoclinic plates, which melt at 118—119°. It is anhydrous, and is readily soluble in water and alcohol, giving an acid solution:

0.2444 gave 0.0834 AgCl. Cl = 8.4.

$\text{C}_{22}\text{H}_{25}\text{O}_4\text{N}\cdot\text{HCl}$ requires Cl = 8.8 per cent.

The *nitrate* separates from alcohol in glistening, diamond-shaped plates, which melt and decompose at 158°. It is anhydrous, and is very sparingly soluble in water, but sparingly so in cold alcohol:

0.1504 gave 0.3384 CO_2 and 0.0810 H_2O . C = 61.4; H = 6.0.

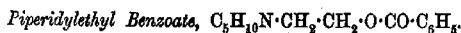
$\text{C}_{22}\text{H}_{25}\text{O}_4\text{N}\cdot\text{HNO}_3$ requires C = 61.3; H = 6.1 per cent.

The *aurichloride* crystallises from alcohol in golden-yellow, monoclinic plates, which melt at 162—163°. It is anhydrous, and is insoluble in water, but sparingly soluble in cold alcohol:

0.2097 gave 0.0585 Au. Au = 27.9.

$\text{C}_{22}\text{H}_{25}\text{O}_4\text{N}\cdot\text{HAuCl}_4$ requires Au = 27.9 per cent.

The *picrate* crystallises from alcohol in yellow, diamond-shaped plates, which melt at 167—168°.



Ten grams of piperidylethyl alcohol gave, on benzoylation, 14.5 grams of piperidylethyl benzoate, a yield of 82 per cent. of the theoretical. The substance was purified by crystallisation of the hydrochloride, and regeneration from the pure salt, when it formed a colourless, viscid oil which did not crystallise.

The *hydrochloride* crystallises from absolute alcohol in large, monoclinic plates, which melt at 175—176°. It is anhydrous, and is easily soluble in water, giving a neutral solution, but sparingly so in alcohol:

0.1782 gave 0.4074 CO_2 and 0.1164 H_2O . $\text{C} = 62.3$; $\text{H} = 7.3$.

0.3116 „ 0.1647 AgCl . $\text{C} = 13.1$.

$\text{C}_{14}\text{H}_{19}\text{O}_2\text{N}$, HCl requires $\text{C} = 62.3$; $\text{H} = 7.5$; $\text{Cl} = 13.1$ per cent.

The *hydrobromide* separates from its alcoholic solution in glistening plates, which melt at $168-169^\circ$. It is anhydrous, and is fairly readily soluble in water, but sparingly so in alcohol:

0.2042 gave 0.1214 AgBr . $\text{Br} = 25.9$.

$\text{C}_{14}\text{H}_{19}\text{O}_2\text{N}$, HBr requires $\text{Br} = 25.4$ per cent.

The *aurichloride* crystallises from alcohol in serrated needles, which melt at $119-120^\circ$. It is anhydrous, and is insoluble in water, but somewhat sparingly soluble in cold alcohol:

0.1226 gave 0.0422 Au . $\text{Au} = 34.4$.

$\text{C}_{14}\text{H}_{19}\text{O}_2\text{N}$, HAuCl_4 requires $\text{Au} = 34.4$ per cent.

The *picrate* crystallises from alcohol in clusters of yellow needles, which melt at $160-161^\circ$.

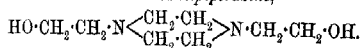
The *methobromide* crystallises from a mixture of alcohol and light petroleum in slender needles, which melt at $144-147^\circ$. It is anhydrous, and is readily soluble in water or alcohol:

0.2138 gave 0.4318 CO_2 and 0.1260 H_2O . $\text{C} = 55.1$; $\text{H} = 6.6$.

0.1050 „ 0.0600 AgBr . $\text{Br} = 24.3$.

$\text{C}_{14}\text{H}_{19}\text{O}_2\text{N}$, CH_3Br requires $\text{C} = 54.8$; $\text{H} = 6.8$; $\text{Br} = 24.4$ per cent.

1:4-Diethanolpiperazine,



Six grams of piperazine and 14 grams of ethylene chlorohydrin were heated together for four hours at 105° . The resulting dark brown oil was made alkaline with 50 per cent. aqueous sodium hydroxide, diluted with 150 c.c. of alcohol, saturated with carbon dioxide, and filtered. The alcoholic filtrate was then dried with anhydrous sodium sulphate, filtered, and distilled. After the removal of the alcohol, the distillation was continued under 50 mm. pressure, when the base passed over at $215-220^\circ$; after crystallisation from absolute alcohol, 5 grams of pure base were obtained; this yield represents 41 per cent. of the theoretical.

The base separates from absolute alcohol in large, tetragonal pyramids, which melt at $134-135^\circ$. It is very readily soluble in water, and readily so in chloroform, but sparingly so in the other usual organic solvents:

0.1702 gave 0.3446 CO_2 and 0.1618 H_2O . $\text{C} = 55.2$; $\text{H} = 10.6$.

$\text{C}_8\text{H}_{18}\text{O}_2\text{N}_2$ requires $\text{C} = 55.1$; $\text{H} = 10.4$ per cent.

The *hydrochloride* crystallises from absolute alcohol in prismatic

plates, which contain two molecules of water of crystallisation. It is very easily soluble in water, but sparingly so in absolute alcohol. After drying at 120°, this salt melts at 200—202°:

0.1940, air-dried, lost 0.0242 at 120°. $H_2O = 12.5$.

0.1628, dried at 120°, gave 0.1886 AgCl. $Cl = 28.7$.

$C_8H_{18}O_2N_2 \cdot 2HCl \cdot 2H_2O$ requires $H_2O = 12.7$ per cent.

$C_8H_{18}O_2N_2 \cdot 2HCl$ requires $Cl = 28.7$ per cent.

The *hydrobromide* crystallises from acetone in highly-refracting, prismatic plates, which contain two molecules of water of crystallisation. It is very readily soluble in water, but sparingly so in alcohol or acetone. After drying at 120°, this salt melts at 190—192°:

0.2622, air-dried, lost 0.0250 at 120°. $H_2O = 9.5$.

0.1494, dried at 120°, gave 0.1674 AgBr. $Br = 47.7$.

$C_8H_{18}O_2N_2 \cdot 2HBr \cdot 2H_2O$ requires $H_2O = 9.7$ per cent.

$C_8H_{18}O_2N_2 \cdot 2HBr$ requires $Br = 47.6$ per cent.

The *nitrate* crystallises from absolute alcohol in diamond-shaped plates, which melt at 177—178°. It is anhydrous, and is readily soluble in water, but sparingly so in absolute alcohol:

0.1968 gave 0.2302 CO_2 and 0.1134 H_2O . $C = 31.9$; $H = 6.5$.

$C_8H_{18}O_2N_2 \cdot 2HNO_3$ requires $C = 32.0$; $H = 6.7$ per cent.

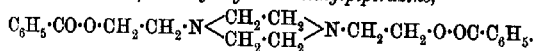
The *aurichloride* separates from very dilute hydrochloric acid in long, light yellow, prismatic needles, which melt and decompose at 205—207°. It is anhydrous, and is sparingly soluble in cold water, but moderately so in alcohol:

0.1240 gave 0.0570 Au. $Au = 46.0$.

$C_8H_{18}O_2N_2 \cdot 2HAuCl_4$ requires $Au = 46.2$ per cent.

The *picrate* crystallises from alcohol in fine yellow needles, which melt and decompose at 245—246°.

s-Di-β-benzoyloxy-1:4-diethylpiperazine,



Three grams of 1:4-diethanolpiperazine gave, on benzoylation, a quantity of solid *s-di-β-benzoyloxy-1:4-diethylpiperazine*; this was collected, washed with water, dried on a porous plate, and purified by crystallisation from absolute alcohol; the yield was 6 grams, that is, 91 per cent. of the theoretical.

The base crystallises from ether in long, prismatic needles, which melt at 104—105°; it is insoluble in water, fairly readily soluble in alcohol, and sparingly so in ether:

0.1998 gave 0.5076 CO_2 and 0.1234 H_2O . $C = 69.3$; $H = 6.9$.

$C_{22}H_{30}O_4N_2$ requires $C = 69.1$; $H = 6.9$ per cent.

The *hydrochloride* separates from boiling water in colourless needles, which melt and decompose at 260°. It is anhydrous, and is very sparingly soluble in cold water or alcohol:

0.1078 gave 0.0667 AgCl. Cl = 15.3.

$C_{22}H_{26}O_4N_2 \cdot 2HCl$ requires Cl = 15.6 per cent.

The *hydrobromide* crystallises from dilute hydrobromic acid in rosettes of feathery needles, which melt and decompose at 264–265°. It is anhydrous, and is very sparingly soluble in cold water or alcohol:

0.1476 gave 0.1006 AgBr. Br = 29.0.

$C_{22}H_{26}O_4N_2 \cdot 2HBr$ requires Br = 29.4 per cent.

The *sulphate* crystallises from water in clusters of glistening needles, which melt and decompose at 219–220°. It is anhydrous, and is very sparingly soluble in cold water or alcohol:

0.1346 gave 0.0662 BaSO₄. SO₄ = 20.3.

$C_{22}H_{26}O_4N_2 \cdot H_2SO_4$ requires SO₄ = 20.0 per cent.

The *aurichloride* crystallises from alcohol in monoclinic plates, which melt and decompose at 205°. It is anhydrous, and is insoluble in water, but very sparingly soluble in alcohol:

0.0818 gave 0.0303 Au. Au = 36.9.

$C_{22}H_{26}O_4N_2 \cdot 2HAuCl_4$ requires Au = 37.1 per cent.

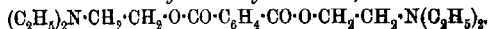
The *picrate* crystallises from methyl alcohol in pale yellow, prismatic needles, which melt and decompose at 265–267°.

The *methobromide* crystallises from water in prismatic needles, which melt at 228–229°. It is very sparingly soluble in cold water, and is insoluble in alcohol:

0.2582 gave 0.1678 AgBr. Br = 27.7.

$C_{22}H_{26}O_4N_2 \cdot 2CH_3Br$ requires Br = 27.9 per cent.

Diethylaminoethyl Phthalate,



Eighteen grams of phthaloyl chloride were diluted with 50 c.c. of anhydrous ether, and added to a solution of 18 grams of β -hydroxytriethylamine in 50 c.c. of anhydrous ether. A white, crystalline precipitate of diethylaminoethylphthalate hydrochloride was immediately produced; this was allowed to stand for half an hour, collected, dissolved in water, and the aqueous solution extracted with ether to remove impurities; the solution was then rendered alkaline with solid sodium carbonate, and extracted with chloroform; on distilling the chloroform, 21 grams of the base were obtained as a colourless, viscid oil, the yield representing 75 per cent. of the theoretical.

The *hydrobromide* crystallises from acetone in bushy crystals, which,

after drying at 100° , melt at $157-159^{\circ}$. It is very readily soluble in water, and fairly readily so in alcohol:

0.1486, dried at 100° , gave 0.2470 CO_2 and 0.0842 H_2O . $\text{C} = 45.3$; $\text{H} = 6.3$.

0.1802, dried at 100° , gave 0.1286 AgBr . $\text{Br} = 30.4$.

$\text{C}_{20}\text{H}_{22}\text{O}_4\text{N}_2 \cdot 2\text{HBr}$ requires $\text{C} = 45.6$; $\text{H} = 6.5$; $\text{Br} = 30.4$ per cent.

The *platinichloride* crystallises from water in clusters of fine needles, which melt at $191-193^{\circ}$. It contains one molecule of water of crystallisation, and is fairly readily soluble in hot water, but sparingly so in cold water:

0.1240, air-dried, lost 0.0030 at 100° . $\text{H}_2\text{O} = 2.4$.

0.1210, dried at 100° , gave 0.0300 Pt . $\text{Pt} = 24.8$.

$\text{C}_{20}\text{H}_{22}\text{O}_4\text{N}_2 \cdot \text{H}_2\text{PtCl}_6 \cdot \text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 2.3$ per cent.

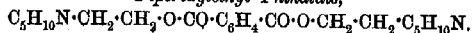
$\text{C}_{20}\text{H}_{22}\text{O}_4\text{N}_2 \cdot \text{H}_2\text{PtCl}_6$ requires $\text{Pt} = 25.1$ per cent.

The *picrate* separates from boiling alcohol in yellow, prismatic crystals, which melt at $146-147^{\circ}$. It is anhydrous, and is very sparingly soluble in water or alcohol:

0.1884 gave 0.3246 CO_2 and 0.0848 H_2O . $\text{C} = 47.0$; $\text{H} = 5.0$.

$\text{C}_{20}\text{H}_{22}\text{O}_4\text{N}_2 \cdot 2\text{C}_6\text{H}_3\text{O}_7\text{N}_3$ requires $\text{C} = 46.7$; $\text{H} = 4.7$ per cent.

Piperidylethyl Phthalate,



This base was prepared in a similar manner to diethylaminoethyl phthalate. Twenty-eight grams of phthaloyl chloride and 36 grams of piperidylethyl alcohol gave 37 grams of the base as a colourless, viscid oil, the yield representing 68 per cent. of the theoretical.

The *hydrochloride* crystallises from a mixture of absolute alcohol and ether in rosettes of long, white needles, which, after drying at 100° , melt at $190-191^{\circ}$. Before drying at 100° , the salt is very deliquescent, and probably contains solvent of crystallisation, but the nature and amount of this were not determined. The salt is very readily soluble in water or absolute alcohol:

0.246, dried at 100° , gave 0.1553 AgCl . $\text{Cl} = 15.6$.

$\text{C}_{22}\text{H}_{28}\text{O}_4\text{N}_2 \cdot 2\text{HCl}$ requires $\text{Cl} = 15.4$ per cent.

The *hydrobromide* crystallises from absolute alcohol in radiating, prismatic needles, which melt at $205-206^{\circ}$ to a red liquid. It is anhydrous, and is very readily soluble in water, but moderately so in absolute alcohol:

0.1638 gave 0.2864 CO_2 and 0.0916 H_2O . $\text{C} = 47.7$; $\text{H} = 6.3$.

0.1968 „ 0.1352 AgBr . $\text{Br} = 29.2$.

$\text{C}_{22}\text{H}_{28}\text{O}_4\text{N}_2 \cdot 2\text{HBr}$ requires $\text{C} = 48.0$; $\text{H} = 6.2$; $\text{Br} = 29.1$ per cent.

The *nitrate* crystallises from absolute alcohol in rosettes of fine

prismatic needles, which melt at 169—170°. It contains one-half a molecular proportion of water of crystallisation, and is very readily soluble in water, and moderately so in alcohol:

0.1896, air-dried, gave 0.3504 CO₂ and 0.1138 H₂O. C = 50.4; H = 6.7.
 0.2646 „ „ lost 0.0048 at 130°. H₂O = 1.8.
 (C₂₂H₃₂O₄N₂, 2HNO₃)₂, H₂O requires C = 50.5; H = 6.8; H₂O = 1.7 per cent.

The *platinichloride* crystallises from water in rosettes of fine reddish-yellow needles, which melt at 205—206°. It contains one molecule of water of crystallisation, and is sparingly soluble in cold water, but very sparingly so in alcohol:

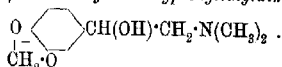
0.0990, air-dried, lost 0.0028 at 140°. H₂O = 2.8.
 0.0962, dried at 140°, gave 0.0236 Pt. Pt = 24.5.

C₂₂H₃₂O₄N₂, H₂PtCl₆, H₂O requires H₂O = 2.2 per cent.

C₂₂H₃₂O₄N₂, H₂PtCl₆ requires Pt = 24.4 per cent.

The *picrate* crystallises from methyl alcohol in fine yellow needles, which melt at 182—183°.

β-Hydroxy-β-3:4-methylenedioxyphenylethyl dimethylamine,



Seventeen grams of *β-bromo-α-hydroxy-3:4-methylenedioxyethylbenzene* were dissolved in 80 c.c. of absolute alcohol, and boiled for two hours under reflux with 50 c.c. of a 33 per cent. aqueous solution of dimethylamine. After distillation of the alcohol, the product was rendered alkaline and extracted with ether. The base was then withdrawn from the ether by dilute hydrochloric acid, regenerated with sodium carbonate, and again dissolved in ether. The ethereal solution gave, on evaporation, 13 grams of base, that is, 90 per cent. of the theoretical.

The base was purified by crystallisation of the *picrate* and regeneration from the pure salt, when it formed a viscid, colourless oil which did not crystallise.

The *hydrochloride* crystallises from absolute alcohol in prismatic needles, which melt at 151—153°. It is anhydrous, and is readily soluble in water or hot alcohol:

0.1512 gave 0.2954 CO₂ and 0.0902 H₂O. C = 53.3; H = 6.7.
 0.1242 „ „ 0.0732 AgCl. Cl = 14.6.

C₁₁H₁₅O₃N, HCl requires C = 53.7; H = 6.6; Cl = 14.4 per cent.

The *aurichloride* crystallises from alcohol in dark brown nodules, which melt at 111—112°. It is anhydrous, and is readily soluble in hot water or alcohol:

0.1768 gave 0.0642 Au. Au = 36.3.

$C_{11}H_{15}O_2N.HAuCl_4$ requires Au = 35.9 per cent.

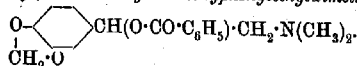
The *picrate* crystallises from water in fine prismatic needles, which melt at 98—100°, and contain one molecule of water of crystallisation. It is readily soluble in hot water :

0.1130, air-dried, gave 0.1858 CO_2 and 0.0452 H_2O . C = 44.8 ; H = 4.5.

0.1446 „ lost 0.0052 at 125°. H_2O = 3.6.

$C_{11}H_{15}O_3N.C_6H_5O_7N_3.H_2O$ requires C = 44.7 ; H = 4.4 ; H_2O = 3.9 per cent.

β-Benzoyloxy-β-3 : 4-methylenedioxyphenylethyldimethylamine,



Five grams of *β*-hydroxy-β-3 : 4-methylenedioxyphenylethyldimethylamine gave, on benzylation, 5 grams of the benzoylated base ; the yield represents 78 per cent. of the theoretical. The substance was purified by crystallisation of the hydrochloride and regeneration from the pure salt, when it formed a colourless, viscid oil which did not crystallise.

The *hydrochloride* crystallises from hot water in glistening prisms, which melt at 216°. It is anhydrous, and is sparingly soluble in cold water or alcohol :

0.1786 gave 0.0716 AgCl. Cl = 9.9.

$C_{18}H_{19}O_4N.HCl$ requires Cl = 10.1 per cent.

The *hydrogen sulphate* crystallises from absolute alcohol in irregular, lustrous plates, which melt at 89—90°. It contains one molecule of water of crystallisation, and is readily soluble in water or absolute alcohol :

0.1082, air-dried, gave 0.1996 CO_2 and 0.0542 H_2O . C = 50.3 ; H = 5.6.

0.1330 „ lost 0.0050 at 100°. H_2O = 3.8.

0.1466, dried at 100°, gave 0.0834 $BaSO_4$. SO_4 = 23.4.

$C_{18}H_{19}O_4N.H_2SO_4.H_2O$ requires C = 50.3 ; H = 5.4 ; H_2O = 4.2 per cent.

$C_{18}H_{19}O_4N.H_2SO_4$ requires SO_4 = 23.3 per cent.

The *platinichloride* was obtained as a granular precipitate which could not be crystallised. It was purified by dissolution in acetone, and precipitated with ether, when it formed a granular precipitate, which melted and decomposed at 151—153°. It is readily soluble in acetone, but insoluble in water, alcohol, or ether :

0.0748, dried at 100°, gave 0.0140 Pt. Pt = 18.7.

$(C_{18}H_{19}O_4N)_2.H_2PtCl_6$ requires Pt = 18.8 per cent.

The *picrate* crystallises from absolute alcohol in rosettes of fine yellow needles, which melt at 184—185°.

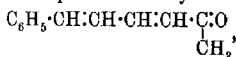
THE WELLCOME CHEMICAL WORKS,
DARTFORD, KENT.

CLXXVIII.—*The Relation between Absorption Spectra and Chemical Constitution. Part X. Unsaturated Acids of the Benzene Series.*

By EDWARD CHARLES CYRIL BALLY and KONRAD SCHAEFER.

AMONG the instances of compounds which do not conform to modern views on the origin of colour, perhaps one of the most striking is that of cinnamylidenemalononic acid. As is well known, the free acid and its esters are yellow, whilst its alkali-metallic salts are quite colourless. It would seem impossible that the replacement of the alkyl groups by a metal should entirely alter the structure of a compound, and yet this is the view that would probably find the greatest favour at the present time. We have examined the absorption spectra of this acid, its methyl ester, and sodium salt, and find that they are strikingly similar, the only difference between them lying in the position of the absorption bands. It is at once evident, if such evidence be needed, that there can be no real difference in constitution between the three substances. The explanation of the shift in the absorption band when the acid is converted into the sodium salt must be found in the fact that the system of conjugated double linkings is readily influenced by the character of the atom replacing the hydrogen of the carboxyl group. In other words, the position of the absorption band must depend on the free residual affinity of the carbonyl portion of the carboxylic groups at the end of the chain.

If we consider the compound cinnamylideneacetone,

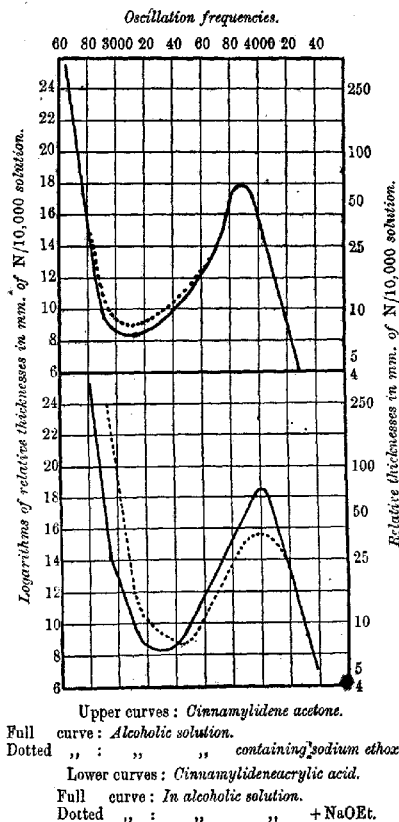


it will be seen that the carbonyl group is conjugated with two double linkings and a phenyl group. The isorropesis due to this conjugation produces the absorption band shown by the full curve in Fig. 1 (upper part), with its head at a frequency of about 3130. In the case of cinnamylideneacetic acid where the methyl group is replaced by hydroxyl, the absorption band, as shown by the full curve in Fig. 1 (lower part), has been shifted towards the violet end of the spectrum, and has now its head at a frequency of about 3320. The free residual affinity of the carbonyl group has been materially altered; a portion has been bound up with the adjacent hydroxyl group, so that there is less free affinity able to conjugate with the system $\text{C}_6\text{H}_5\text{:CH:CH:CH:CH:}$, with the natural sequence that the absorption band of the acid is nearer the violet end of the spectrum than in the case of the ketone. It can be seen at once

ABSORPTION SPECTRA AND CHEMICAL CONSTITUTION. 1809

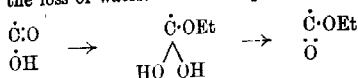
that in these unsaturated acids of the type of cinnamylidenesacetic we are able, from a determination of their absorption spectra, to examine the carboxyl group and estimate relatively the amount of free residual affinity possessed by the carbonyl group. The system

FIG. 1.



formed by the phenyl group with a side-chain containing ethylenic linkings is a very delicate touchstone, against which one may try various atomic groupings with a view to determining their value as centres of residual affinity relatively to some standard. In the

present case, the standard substance is the corresponding ketone. Among the many fields of research at the present time, the carboxyl group and its esterification possesses very considerable interest, both from the point of view of steric hindrance and also from the point of view of catalysis. The actual mechanism of esterification is imperfectly understood, although the most generally accepted view is that the reaction is one of addition of the alcohol followed by the loss of water. For example:

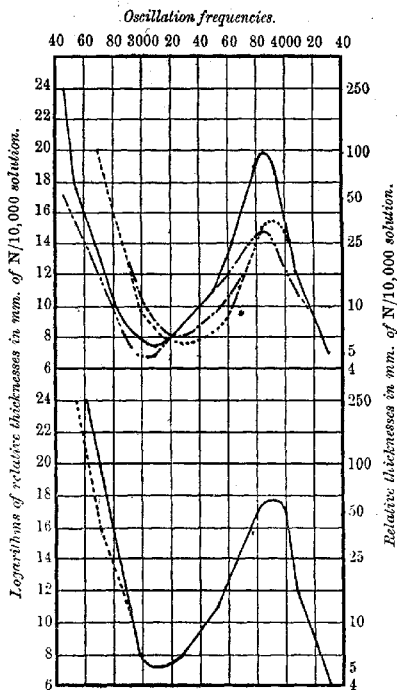


According to this theory the esterification constant of an acid must depend directly on the free residual affinity of the carbonyl group; the greater this free affinity the more readily will the alcohol attach itself to give the intermediary additive compound. This view was put forward in a previous paper (Stewart and Baly, Trans., 1905, **89**, 489), and it was suggested that the activity of the carbonyl group is determined by the nature of the adjacent carbon atom. From analogy to the ketones, the activities of the carbonyl groups should form a descending series in formic, acetic, propionic, and isobutyric acids, and therefore the esterification constant should fall, which, indeed, is a well-known fact. No spectroscopic method is at present available for the determination of the activity or free affinity of the carboxylic carbonyl group in the fatty acids.

The results, however, given by the unsaturated acids of the benzene series show that a second contributory variable lies in the hydroxyl group, and that the activity of the carbonyl group depends to a considerable extent on the character of the hydroxyl group, its ionisation, etc. Fig. 1 (lower curves) shows the absorption of cinnamylideneacetic acid and also of its sodium salt, and in the latter case the absorption band is shifted very distinctly towards the violet end of the spectrum, showing that the carbonyl group has even less free residual affinity than it has in the free acid. In order to investigate more fully the influence of substitution, we examined cinnamylidenemalonic acid, which shows the effect more plainly. The curves obtained are given in Fig. 2 (upper part), the effect of alkali and mineral acid being most marked. The addition of alkali decreases, whilst the addition of acid increases, the free affinity of the carbonyl group, since the absorption band shifts towards the blue in the one case and towards the red in the other. It will also be noticed that a solution of the acid in water develops its absorption band nearer the blue than in alcoholic solution. The natural deduction from this is that the

more the substance is ionised the less free affinity is possessed by the carbonyl group. The affinity is increased when the ionisation is hindered by the addition of hydrogen ions, and decreased in the easily ionisable sodium salt. The explanation of this follows simply

FIG. 2.



Upper curves : Cinnamylidenemalononic acid.

Full curve : Alcoholic solution.

Dotted „ : „ + NaOEt.

Dash and two dots curve : „ Alcoholic solution + HCl.

Dash and dot „ : Aqueous solution.

Lower curves : Methyl cinnamylidenemalonate.

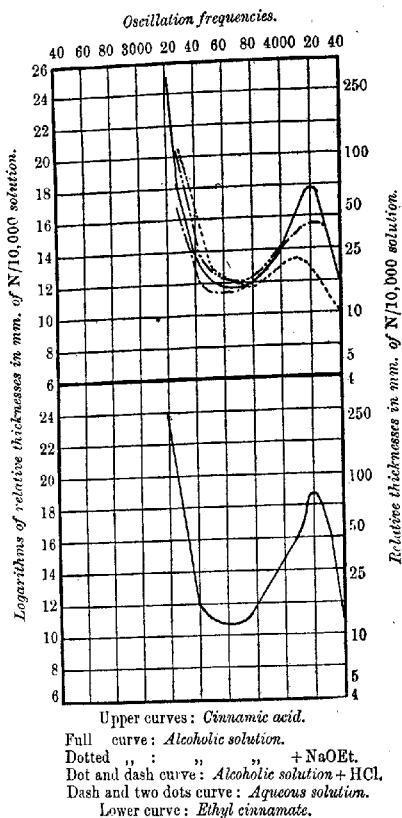
Full curve : Alcoholic solution.

Dotted „ : Aqueous „

from the comparison already drawn between cinnamylideneacetone and cinnamylideneacetic acid. The replacement of the methyl group by the hydroxyl group causes a certain amount of the free affinity of the carbonyl group to be bound up with the hydroxylic

oxygen. The greater the amount of ionisation the greater will be the affinity of the hydroxylic oxygen atom, and hence the greater amount of the affinity of the carbonyl group will be bound up with it, with the result that the carbonyl group will have less free affinity.

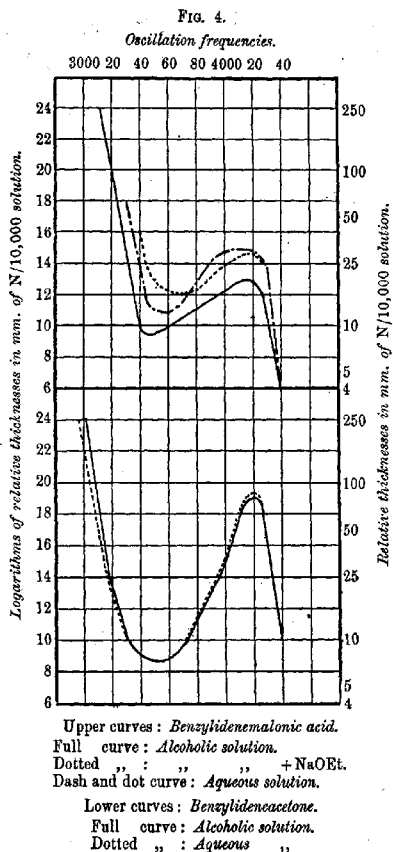
FIG. 3.



Similarly, the less the ionisation the greater the free residual affinity of the carbonyl group.

These results have been confirmed in benzylidenemalononic and cinnamic acids, the absorption spectra of which are shown in Figs. 3 and 4. The absorption spectrum of benzylideneacetone is given in Fig. 4, and may be compared with that of cinnamic acid.

In order to prove that the effects observed with the acids are due to the hydroxyl group only, we have examined benzylideneacetone, cinnamylideneacetone, methyl cinnamylidenemalonate, and ethyl cinnamate under the same conditions, namely, in neutral alcoholic



and aqueous solutions, and also in presence of mineral acid and alkali. Only very minor changes were observed, as are shown in the respective diagrams. In the case of ethyl cinnamate, all the four curves are the same; the aqueous solutions of methyl

cinnamylidenemalonate and benzylidenemalonate show an extremely small variation from the alcoholic, whilst a slight shortening of the band is observed on the addition of alkali to cinnamylidenemalonate. The changes in the position of the absorption band observed with the free acids must therefore be due to the changes in the hydroxyl group.

There are thus two influences at work which determine the affinity of the carbonyl group in carboxylic acids, namely, (1) the nature of the adjacent carbon atom, and (2) the condition of the hydroxylic oxygen. On both of these does the affinity, and hence the esterification value, depend. It may be pointed out that these results throw some light on the catalytic action of mineral acid on the esterification process, because, owing to the probable action of mineral acid in depressing the ionisation of the organic acid, the free affinity, and thus the activity of the carbonyl group, is enhanced; the velocity of esterification is therefore increased.

An interesting and important point arises in connexion with cinnamylidenemalonate. The free acid is yellow, the colour being due to the absorption band with its head at 3100; the sodium salt, since the absorption band is shifted just out of the visible region, appears colourless to the eye. From a physical point of view, it is almost as strongly coloured as the acid, since the absorption band is nearly as deep, but, owing to the fact that the eye happens to be incapable of appreciating the colour, it is considered as colourless. This fact emphasises the danger of forming any theory as to the constitution of any substance from the merely physiological estimate of its colour.

Conclusions.

1. Unsaturated ketones of the type of cinnamylidenemalonate exhibit a well-marked absorption band arising from the isorropesis due to the conjugation of the carbonyl group with the double linkings and the phenyl group.

2. In the unsaturated acids of the type of cinnamylidenemalonate, the absorption band is shifted towards the violet end of the spectrum, because, owing to a certain amount of the residual affinity of the carbonyl group being bound up with the hydroxyl group, there is less free affinity to conjugate with the double linkings and the phenyl group.

3. The amount of the free affinity of the carbonyl group is greatest when the acid is not ionised, and least in the easily ionised sodium salt.

4. The esterification constant of any acid depends on the free

affinity of the carbonyl group; this free affinity depends on two variables, namely, (1) the nature of the atoms or groups united with the adjacent carbon, and (2) the nature of the atom combined with the hydroxylic oxygen.

5. The catalytic action of mineral acid in esterification depends on the fact that mineral acid depresses the ionisation of the organic acid and increases the free affinity of the carbonyl group.

The authors wish to express their thanks to Mr. W. N. Morley, who prepared many of the compounds described in this paper, and also to the Research Fund Committee of the Chemical Society for a grant in aid of the above investigation.

SPECTROSCOPIC LABORATORY,
UNIVERSITY COLLEGE, LONDON.

CLXXIX.—*The Relation between Viscosity and Chemical Constitution. Part II. The Existence of Racemic Compounds in the Liquid State.*

By ALBERT ERNEST DUNSTAN and FERDINAND BERNARD THOLE.

THE marked delicacy of the viscosimetric method renders it extremely probable that valuable information is to be obtained by its application to such problems as that indicated in the title. Of only one previous communication can it be fairly said that a definite answer has been given to the question as to the existence of racemic compounds in solution. Stewart (Trans., 1907, 91, 1537), by the determination of the absorption spectra of aqueous solutions of dextro-, meso-, and racemic tartaric acids, conclusively demonstrated that in the last case a curve was obtained which for low concentrations was identical with that afforded by the optically active forms, but at concentrations above 14 per cent. the curve for racemic acid diverged from that of the dextro-tartaric acid: this observation being indicative of the fact that, as the solution became more concentrated, the racemic form was more and more stable in the dissolved state, whereas at less concentrations than 14 per cent. an almost complete dissociation ensued, a conclusion in agreement with the statements of Raoult (*Zeitsch. physikal. Chem.*, 1887, 1, 186), Ostwald (*J. pr. Chem.*, 1885, [ii], 32, 341), and Marchlewski (*Ber.*, 1892, 25, 1556).

Stewart (*Stereochemistry*, p. 41) points out that, with the above-mentioned exception, there is no method available for the decision

of this question, although Bruni and Padoa (*loc. cit.*) considered that from determinations of molecular weight they had obtained indications of the existence of complex molecules, whilst, on the other hand, Walden finds that racemates in solution have the same affinity constants as the optically active forms, and from vapour density determinations the same identity is discovered.

In a recent paper, Ranken and Taylor (*Proc. Roy. Soc. Edin.*, 1907, 27, 172), on investigating the physical properties of solutions containing independent optically active substances, point out that there is an unmistakable difference of properties between the optically active and the racemic solutions. This difference is most pronounced in the more concentrated solutions, and is no doubt an indication of the existence of racemic ions or molecules in the solution. This conclusion is supported by the fact that in every case the viscosity of the racemic solution is less than that of the optically active form. This observation is of interest because the same authors similarly determine the density of an 8.333 per cent. solution of the dextro-form at 15°C as 1.0370 and of the racemic form at the same temperature and concentration as 1.03712, and Marchlewski gives for similarly comparable solutions of 14.018 per cent. as 1.06600 and 1.06623 respectively. Ranken and Taylor show that, on the average, the racemic solution has a lower density, although it is fair to mention that these authors have not pursued their investigation from the point of view of a quantity which they only determined incidentally.

EXPERIMENTAL.

In the present communication, we have determined the viscosities and densities of dextro-, levo-, meso-, and racemic tartaric acids at 25° through a range of concentration in aqueous solution; dextro- and racemic malic acids in aqueous solution; dextro-, levo-, and racemic limonenes in the liquid state, and levo-amylic alcohol in the liquid state.

The description of apparatus and method will be found in our previous papers (*Trans.*, 1904, 85, 817; 1905, 87, 1; 1907, 91, 83; 1907, 91, 1728).

Most of the materials were specially prepared for us by Schuchardt and Kahlbaum, and were purchased with a grant from the Research Committee of this Society, to whom we make this grateful acknowledgment. We also desire to express our indebtedness to Dr. Stewart for the specimens of amylic alcohol and mesotartaric acid with which he supplied us.

*The Tartaric Acids.**d-Tartaric Acid (Kahlbaum).* M. p. 170°.

Per cent. tartaric acid.	Time of flow in seconds.	Density, 25°/4°.	Viscosity.
0.0	737.2	0.9972	0.00891
4.84	799.4	1.0181	0.00991
7.99	849.7	1.0337	0.01065
11.675	922.2	1.0511	0.01175
15.01	1000.4	1.0673	0.01294
0.0	25.2	0.9972	0.00891
7.69	28.6	1.0328	0.01047

* Thorpe and Rodger.

l-Tartaric Acid (Kahlbaum).

7.69	28.6	1.0328	0.01047
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Mesotartaric Acid.

0.0	282.3	0.9972	0.00891
9.966	339.9	1.0428	0.01120

r-Tartaric Acid (Kahlbaum). M. p. 206°.

0.0	737.2	0.9972	0.00891
4.02	787.2	1.0153	0.00969
7.00	833.7	1.0290	0.01040
13.14	949.3	1.0583	0.01218
0.0	282.3	0.9972	0.00891
15.98	339.4	1.0719	0.01310

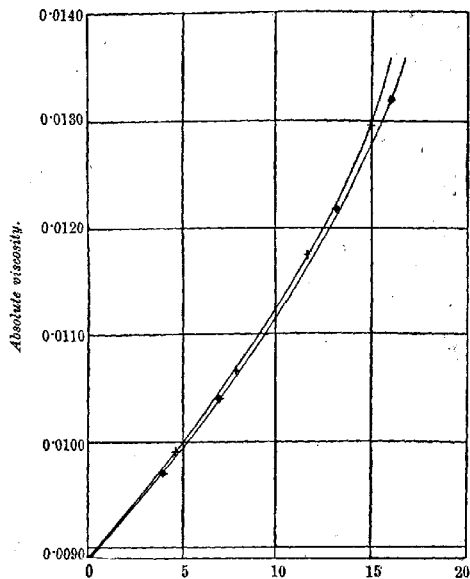
The above results are plotted in Fig. 1, with the exception of that for the meso-acid, which falls near to the curve for the dextro-acid. The same viscometer has been used for each series of determinations to avoid errors arising from calibration. An examination of the figure will show that the curve for the racemic modification lies very uniformly below that of the active form, but it will be noticed that the differences are very small, comparable in fact with those of Ranken and Taylor. Were it not for the low solubility of the racemic acid, it would have been possible to push the observations to a greater concentration, and so probably obtain greater divergences from the curve for the dextro-acid.

The densities of the respective solutions show minute differences, but when plotted on a scale of sufficient size for reproduction, they are found to lie practically on a straight line. As mentioned in the introduction, a degree of accuracy much greater than is justifiable in the present case would be needed to show any marked differences between the racemic and active isomerides.

l-Amyl Alcohol and the Racemic Modification Prepared from it by
Distillation over Potassium Hydroxide. B. p. 129°.

	Time of flow.	Density, 25°/4°.	Viscosity.
Water	282.3	0.9972	0.00891
<i>l</i> -Amyl alcohol.....	1788.0	0.8182	0.04476
<i>r</i> -Amyl „	1588.0	0.8188	0.4115

FIG. 1.



Concentration in grams per 100 grams of solution.
d-Tartaric acid (upper curve) and racemic acid (lower curve).

l- and d-Limonenes (Schuchardt), and the dl-Limonene prepared
therefrom. B. p. 174.5°.

	Time of flow.	Density, 25°/4°.	Viscosity.
Water	282.3	0.9972	0.00891
<i>d</i>	344.8	0.8457	0.00923
<i>l</i>	649.8	0.8782	0.01806
<i>d</i> + <i>l</i>	448.6	0.8617	0.01223
Calculated for <i>d</i> + <i>l</i>	—	—	0.01365

Although when one compares the densities and viscosities of the
individual isomerides some doubt may be expressed as to their chemical

purity, yet the fact that the viscosity of the *d* + *l*-form is less than the calculated mean value is indicative of the formation of a racemic compound.

We also examined specimens of the pinenes, prepared by Schuchardt, here, again, the *d*- and *l*-forms had different viscosities, but the mean value calculated from these values and that determined for the *d* + *l*-form differed.

l- and *d*-Pinenes (Schuchardt). B. p.: dextro = 155°; lævo = 159°.

	Time of flow.	Density, 25°/4°.	Viscosity.
Water	282.3	0.9972	0.00891
<i>d</i>	582.6	0.8711	0.01606
<i>l</i>	518.0	0.8612	0.01412
<i>d</i> + <i>l</i>	555.2	0.8668	0.01523
Calculated for <i>d</i> + <i>l</i>	—	—	0.01509

d- and *r*-Malic Acids (Schuchardt). M. p.: *d* = 98—99°; *r* = 128—129°.

	Time of flow.	Density, 25°/4°.	Viscosity.
Water	282.3	0.9972	0.00891
<i>d</i> , 17.43 per cent.	460.1	1.0665	0.01550
<i>r</i> , 17.43 „	395.6	1.0666	0.01333

The Viscosity of Sodium Ammonium Tartrate Solutions and the Determination of its Transition Point.

Early in the study of stereochemistry the observation was made that sodium ammonium tartrate and sodium ammonium racemate can only exist within definite temperature limits. When Pasteur neutralised racemic acid by equivalent quantities of sodium hydroxide and ammonia, he obtained, on crystallisation, equal amounts of the *d*- and *l*-salts, whereas Stædel, under apparently similar conditions, obtained the sodium ammonium racemate.

Scacchi, in a comprehensive research, showed that at higher temperatures the racemate crystallised, but at lower temperatures the *d* + *l*-tartrates appeared. Wyrnoff further pointed out that 28° was the transition point in question, and that the change involved the absorption or elimination of water, for if powdered sodium ammonium racemate is mixed with water below this temperature, it sets to a solid mass of the mixed *d*- and *l*-tartrates, which apparently melts at 28°.

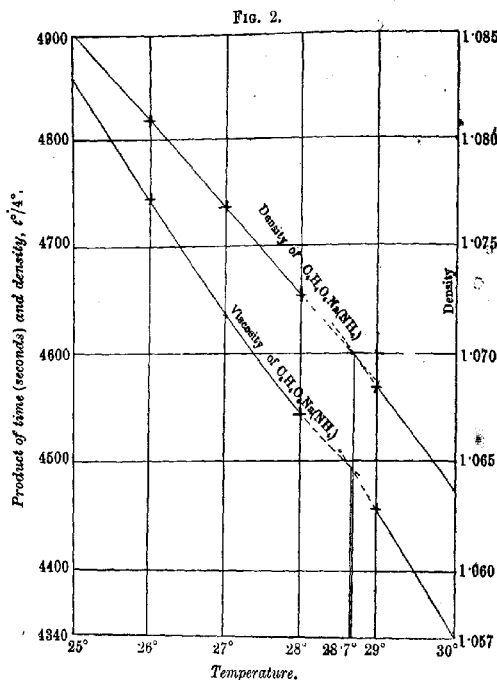
Van't Hoff (*Bildung und Spaltung von Doppelsalzen*, Leipzig, 1897) gives the methods of determining this transition point, namely: dilatometric (rising temperature), 26.7—27.7°; intersection of solubility curves, 27°; dilatometric (falling temperature), 29°.

To supplement the above work from another point of view, and with the expectation of adding yet another method of determining such transition points, we have measured the viscosities and densities

1820 RELATION BETWEEN VISCOSITY AND CHEMICAL CONSTITUTION.

of aqueous solutions of sodium ammonium tartrate (*dI*) at degree intervals from 25° to 30°.

To avoid calibration errors of the viscometer, we have taken the product, time of flow \times density, as being equivalent to viscosity.



Viscosity and density of sodium ammonium racemate and tartrate.

Sodium ammonium tartrate (Schuchardt):

0.6934 lost 0.1959 H_2O at 110°. $H_2O = 28.2$.

$C_4H_8O_6.NNa.4H_2O$ requires $H_2O = 28.0$ per cent.

One hundred grams of solution contained 25.19 grams of the crystallised salt.

Temp.	Time of flow.	Density.	$t \times d$.
25*	433.3	1.085	485.9
26	423.2	1.081	474.4
27	418.4	1.077	464.5
28	410.3	1.072	454.3
29	402.6	1.068	445.6
30	392.3	1.064	434.0

The above results are plotted in Fig. 2. It is to be noticed that each figure shows two curves intersecting at 28.7° respectively for viscosity and density curves. The dotted parts of the curve were not, of course, determined, but serve to show the normal continuations of the full curves. It is probable that each curve shades into the other gradually from both sides of the transition point.

We intend to pursue this side of the work more completely in a further paper.

The following conclusions may be summarised :

- I. The viscosities of *d*- and *l*-forms of tartaric acid are identical.
- II. Mesotartaric acid has a viscosity very close to that of the *d*-form.

III. Racemic compounds exist in solution, but at small concentrations dissociation takes place to a varying extent. It seems likely that the racemic compound has less residual affinity than the active forms ; hence the increased viscosity due to the increased (doubled) molecular weight is more than balanced by the diminution due to a less degree of homogeneous or heterogeneous association.

On the other hand, it may of course happen that the formation of a more symmetrical molecular grouping favours mobility.

IV. The transition point between a racemate and its antipodes may be demonstrated by determining the viscosity through a range of temperature, when a change of curvature occurs at the transition point. The same point is also shown on the density curve, but to a less marked extent.

The authors desire to thank Assistant-Professor Smiles and Dr. Stewart for their suggestions and criticism.

PHYSICAL CHEMICAL LABORATORY,
EAST HAM TECHNICAL COLLEGE.

CLXXX.—*The Direct Interaction of Magnesium and Alkyl Halides.*

By JAMES FREDERICK SPENCER and MARY S. CREWDSON.

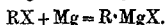
IN a previous paper (Spencer and Stokes, *Trans.*, 1908, 93, 68), it has been shown that aryl bromides and iodides react directly with magnesium when heated ; it had not, however, been observed at the time that aryl chlorides or alkyl halides react with magnesium under the conditions employed in the experiments in question. Löhr

(*Annalen*, 1891, 261, 72) has shown that methyl iodide reacts with magnesium when the two substances are heated together in the presence of a small quantity of ethyl acetate in a sealed tube. Ethyl and propyl iodide also react with magnesium when heated in sealed tubes, giving mixtures of saturated and unsaturated hydrocarbons, together with small quantities of organo-magnesium compounds. The present communication shows that both aryl chlorides and alkyl halides will react directly with magnesium when they are heated together under suitable conditions.

Aryl chlorides, and all the lower alkyl halides up to the butyl derivatives, only react with magnesium when heated to about 270° in a sealed tube for several hours.

Alkyl halides higher in the series than the butyl derivatives react with magnesium when they are heated with it at their boiling point for a few minutes. The reaction is regarded by the authors as taking place along the two lines indicated.

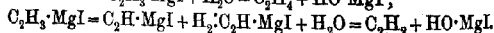
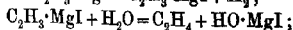
1. The formation of a magnesium alkyl halide, thus:



2. The formation of a hydrocarbon containing twice as many carbon atoms as the original halide, thus:



In addition to these compounds, however, unsaturated hydrocarbons are also found in the gaseous portion of the reaction products obtained from all interactions effected in sealed tubes. These unsaturated hydrocarbons, consisting as they do of both acetylenes and olefins, are due in all probability to the action of heat on the original halide, or on the initial products of the reaction, for Miss Kahan⁴ (*Trans.*, 1908, 93, 133) has shown that methyl and ethyl iodides are decomposed by heat with the formation of unsaturated hydrocarbons. Saturated hydrocarbons are not the only products when water is added to the solid product obtained by the action of an alkyl halide on magnesium in a sealed tube. In addition to the saturated hydrocarbon expected, unsaturated hydrocarbons, consisting of both acetylenes and olefins, are also formed. The presence of these compounds in the product can be explained if we assume that the magnesium alkyl halide first formed has been decomposed by the action of heat with the formation of an unsaturated organo-magnesium halide and free hydrogen, as indicated by the equations:



The invariable occurrence of large quantities of hydrogen in the gaseous products can be regarded as confirmation of this assumption.

In all reactions where it was unnecessary to use a sealed tube, unsaturated hydrocarbons were not found among the products.

The magnesium alkyl halides are all white, crystalline compounds, and are produced in larger quantities the higher in the series the corresponding hydrocarbon stands; thus *sec*-octyl iodide furnishes a magnesium compound, which, on treatment with water, gives 85 per cent. of the theoretical yield of octane, whilst *iso*amyl iodide gives 60 per cent. of the theoretical quantity of pentane. Halogen substituted compounds, other than the hydrocarbon derivative, also react directly with magnesium; for example, methyl bromoacetate reacts very readily with magnesium on heating to its boiling point. We should like to point out here that, while this work was proceeding, and after the reaction with methyl bromoacetate had been completed, we received a copy of a paper from J. B. Tingle and E. E. Gorsline (*J. Amer. Chem. Soc.*, 1907, 37, 483), acquainting us with their work on the Grignard reaction; this paper contained an account of the influence of solvents on the ease with which the reactions take place. These investigators draw our attention especially to a reaction between magnesium and ethyl chloroacetate in ether and other solvents, and mention in their letter that they have also induced this reaction to take place without solvents, presumably, since it is stated neither in their letter nor the paper cited, with the formation of ethyl acetate. In our experiments, methyl acetate was one of the products, but dimethyl succinate was also formed, and to a much larger extent.

EXPERIMENTAL.

In all experiments where sealed tubes were not used, the method of carrying out the reaction was exactly the same as that adopted in the paper already quoted.

In those reactions which only proceeded in sealed tubes, the theoretically proportionate quantities of the halide and magnesium were sealed up, and heated at about 250° for from five to eight hours. When the tubes were cold, they were immersed in liquid air and opened; generally there was no pressure on opening when the tubes were treated in this manner, a fact which would indicate the absence of any large quantity of hydrogen. The tube was then removed from the liquid air, and connected with a gas holder, in which the evolved gas collected as the tube became warmer. This gas invariably contained large quantities of hydrogen, a fact which suggests that magnesium possesses the property of absorbing hydrogen at low temperatures. We are at present investigating this point. The gas was analysed in the usual way. After the tube had attained the atmospheric tempera-

ture, and no more gas was evolved, the solid residue was treated with water, and the gas or liquid evolved was collected and analysed.

Interaction of Magnesium and Methyl Iodide.

Ten grams of methyl iodide and 1.75 grams of dry powdered magnesium were heated in a sealed tube for seven hours at 280°. An inflammable gas was obtained, which consisted of 56 per cent. of hydrogen, 42 per cent. of ethane, 1 per cent. of acetylene, and 0.5 per cent. of ethylene. On treating the solid in the tube with water, a gas was obtained which was inflammable, burning with a smoky flame, and consisting of 72.6 per cent. of hydrogen, 9.2 per cent. of methane, 13.6 per cent. of acetylenes, and 3.3 per cent. of olefines.

Interaction of Magnesium and isoAmyl Iodide.

Ten grams of isoamyl iodide were heated in a hard glass flask with 1.5 grams of magnesium for about half a minute, when a violent reaction set in; this completed itself without further heating, and a white, microcrystalline substance was formed, which, on the addition of water, yielded 62 per cent. of the theoretical quantity of isopentane.

Interaction of Magnesium and sec.-Octyl Iodide.

Ten grams of sec.-octyl iodide were heated in a hard glass flask with one gram of magnesium for a few seconds, when a violent reaction commenced and proceeded to completion without further heating. The product of this interaction was a white, crystalline compound, which reacted violently with water, producing an oil. The oil was separated and fractionated, and constituted a yield of 84 per cent. of octane. The intermediate compound obtained in this experiment was purified and analysed in the following manner: it was washed twice with cold ether to remove any unchanged octyl iodide, then with warm ether to separate the compound from unchanged magnesium, the ethereal solution evaporated to dryness, and the product heated to 100° to decompose any double compound with ether. In this way, a white, crystalline substance was obtained in long, rhombic needles, which were quickly weighed, treated with water and dilute nitric acid, and evaporated to dryness; the magnesium was estimated as pyrophosphate:

0.1037 gave 0.0420 $\text{Mg}_2\text{P}_2\text{O}_7$. $\text{Mg} = 8.78$.

$\text{C}_8\text{H}_{17}\text{IMg}$ requires $\text{Mg} = 9.08$ per cent.

This compound dissolves in ether with extreme readiness.

Interaction of Magnesium and Ethyl Chloride.

Ten grams of ethyl chloride and 4 grams of magnesium were heated together for six hours in a sealed tube at 260°. The gaseous products were found to consist of 45 per cent. of butane, 20 per cent. of olefines, and 22 per cent. of hydrogen. The yield of these gases was large, about 350 c.c. of the mixture being collected. On adding water to the white solid remaining in the tube, an inflammable gas was evolved, which consisted of 86 per cent. of hydrogen and 13 per cent. of ethane; there were no unsaturated hydrocarbons formed in this experiment.

Interaction of Magnesium and isobutyl Chloride.

Ten grams of isobutyl chloride were heated with 2.4 grams of magnesium in a sealed tube at 260° for seven hours. On opening the tube, only a small quantity of gas was evolved, and this, on analysis, proved to be practically all hydrogen; there was, however, in the tube a considerable quantity of a liquid; this was removed from the solid product by draining, and was found to consist of unchanged isobutyl chloride and diisobutyl, boiling at 108°; 2.5 grams of the latter were obtained, which constituted a yield of 40 per cent. The solid residue in the tube was then treated with water, and the gas evolved, collected, and analysed; it consisted of 58 per cent. of hydrogen, 18 per cent. of butane, 11.3 per cent. of acetylenes, and 11.6 per cent. of olefines.

Interaction of Magnesium and Ethyl Bromide.

Twelve grams of ethyl bromide were heated together with 2 grams of magnesium in a sealed tube for eight hours at 260°. The gaseous products consisted of 56 per cent. of hydrogen, 12 per cent. of olefines, 7 per cent. of acetylenes, and 24 per cent. of butane. The white compound left in the tube, on treatment with water, gave an inflammable gas, consisting of 90 per cent. of hydrogen, 6 per cent. of ethane, 2.5 per cent. of acetylenes, and 1 per cent. of olefines.

Interaction of Magnesium and isoAmyl Bromide.

Ten grams of isoamyl bromide and 1.6 grams of magnesium were heated together in a small, hard glass flask until the reaction commenced, that is, for about three minutes. The reaction was not very violent, but nevertheless it completed itself without further heating. The product was a light, bulky, white powder, together with an oil; the whole was treated with water, when the usual violent reaction

set in. The mixture was distilled in a current of steam, and the oil dried and fractionated. It was found to consist of a small quantity of isopentane, and a 67 per cent. yield of *diisopentyl*, boiling at 150°.

Interaction of Magnesium and Methyl Bromoacetate.

Twenty-one grams of methyl bromoacetate were heated with 3 grams of magnesium in a hard glass flask for about a minute, when an extremely violent reaction took place. After the products had cooled, water was added, and, again, a violent reaction took place. The whole was now distilled in a current of steam, and the distillate was separated, dried, and fractionated. Forty-one per cent. of the bromoacetate was regained unchanged, and two other fractions were also obtained. The first, boiling at 57–63°, was methyl acetate, and constituted a 34 per cent. yield on the bromoacetate which had reacted, and the second, boiling at 190–200°, was dimethyl succinate, the yield being 48 per cent. This compound, which on redistillation boiled at 197°, was hydrolysed by boiling with potassium hydroxide, and the potassium salt was converted into the barium salt by treatment with barium hydroxide solution. The barium salt was analysed by converting it into barium sulphate:

0.2084 gave 0.1943 BaSO_4 . Ba = 54.86.

$\text{C}_4\text{H}_6\text{O}_4\text{Ba}$ requires Ba = 54.2 per cent.

Interaction of Magnesium and Chlorobenzene.

Ten grams of chlorobenzene were heated in a sealed tube with 2.5 grams of magnesium for six hours at 270°. On opening the tube, there was no evolution of gas, but, mixed with a somewhat charred mass having a strong odour of benzene, were white crystals. The whole, on treatment with water, became very hot, and benzene was liberated in fairly large quantities, the average yield being 60 per cent. We are proposing to pursue this reaction further.

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CLXXXI.—*The Interaction of Metals of the Aluminium Group and Organic Halogen Derivatives.*

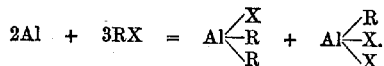
By JAMES FREDERICK SPENCER and MARION L. WALLACE.

THE case with which magnesium reacts with organic halogen derivatives suggested that other metals might also react with these compounds.

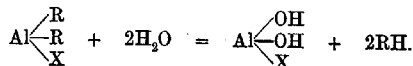
The present paper describes a series of experiments in which the action of aluminium, indium, and thallium on organic halogen derivatives has been studied. Cahours has shown (*Jahresb.*, 1873, 522) that aluminium combines with methyl iodide and ethyl iodide when heated to 100–130°, with the formation, presumably, of the respective aluminium alkyl iodides.

Aluminium in a finely-divided condition reacts readily with halogen derivatives of the hydrocarbons, and, although intermediate additive compounds, similar to those obtained in the case of magnesium, have not been obtained, yet the action of water on the products allows us to conclude that such compounds are present.

The action can be indicated as taking place as follows:



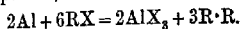
These compounds then react with water, and yield the parent hydrocarbon, thus:



The interaction between aluminium and organic halogen derivatives differs from the corresponding reaction with magnesium (*Trans.*, 1908, 93, 68), inasmuch as it often requires prolonged heating before it commences, and then in some cases proceeds with almost explosive violence. In several experiments, for example, that in which aluminium and *n*-bromonaphthalene were heated together, there was no evolution of heat when water was added to the product; the reason for this is probably that, the reaction being so slow, there is sufficient time for the atmospheric moisture to decompose the intermediate compound as quickly as it is formed, as is known to be the case with magnesium alkyl halides when they are allowed to stand for a short time in the air. This would also explain the large yields of the less volatile hydrocarbons, for example, naphthalene from bromonaphthalene, and the small yield of the volatile hydrocarbons, for example, benzene from iodobenzene. On the above hypothesis, an intermediate com-

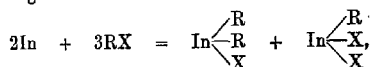
pound would be formed in both cases, which would be decomposed as quickly as formed, giving naphthalene and benzene respectively; the naphthalene being relatively non-volatile would remain, but the benzene, being so very volatile, would disappear in the course of twenty-hours' heating. The chloro-derivatives, with the exception of the chloroanilines, only react when heated in a sealed tube; the aliphatic derivatives lower in the series than the butyl derivatives also react only when heated in a sealed tube, but those higher in the series than the butyl derivatives react when they are heated together with aluminium in an open flask. This is similar to their action with magnesium (compare the preceding paper, p. 1821).

Aluminium does not seem to favour the formation of compounds containing twice as many carbon atoms as there were present in the original halogen compounds, as is the case with magnesium (*loc. cit.*).



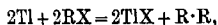
Only in the case of the substituted anilines could any indication of this reaction be obtained, and here small quantities of benzidine were obtained.

Both indium and thallium were found to react slowly with α -bromo-naphthalene and iodobenzene, giving the corresponding hydrocarbons, naphthalene and benzene respectively. The marked slowness of the reaction in the case of these two metals can be attributed to the coarse state of subdivision of the metals. In neither case was the addition of water accompanied by the evolution of heat, but the presence of hydrocarbons in the product allows us to assume that the reaction takes place along the same lines as in the case of aluminium:



the intermediate compounds then being decomposed by the atmospheric moisture.

Thallium gave indications of a second reaction taking place:



for with iodobenzene a small yield of diphenyl was obtained.

Preliminary experiments were made on the action of lithium and other metals on organic halogen derivatives, and it is our intention to continue these experiments. It may be noted, however, that lithium gives a 40 per cent. yield of naphthalene by its action on α -bromo-naphthalene. None of the metals mentioned in this paper could be made to react with organic halogen derivatives in ethereal solution, as is the case with magnesium.

EXPERIMENTAL.

Interaction of Aluminium and Methyl Iodide.

Five grams of methyl iodide and one gram of finely-powdered aluminium were heated together in a sealed tube at 260° for six hours. The tube, after cooling, was placed in liquid air, and then opened; it was then removed from the liquid air and connected with a gasholder, and allowed to attain the atmospheric temperature. A large quantity of an inflammable gas was evolved, which was found to consist of 48 per cent. of hydrogen and 52 per cent. of ethane. The solid product left in the tube, consisting of a white, crystalline compound and unchanged aluminium, was treated with water, when it evolved an inflammable gas. This consisted of about 82 per cent. of hydrogen, and 18 per cent. of methane, together with small quantities of ethylene and acetylene.

*Interaction of Aluminium and isoAmyl Iodide.**

Five grams of isoamyl iodide and 0.7 gram of powdered aluminium were mixed in a small, hard glass flask, fitted with a condenser, and gently heated for about one minute, when a violent reaction took place, hydrogen iodide being liberated. When cold, the contents of the flask were treated with water; a reaction took place with the evolution of heat, and the formation of an inflammable gas and a small quantity of an oil. The oil was isopentane, b. p. 30° , and the gas consisted of 32 per cent. of isopentane, 66 per cent. of hydrogen, and 2 per cent. of olefines.

Interaction of Aluminium and isoAmyl Bromide.

Five grams of isoamyl bromide and 0.8 gram of aluminium were mixed in a small flask, fitted with a condenser, and gently heated; after about twenty minutes a violent reaction took place with the evolution of hydrogen bromide. On cooling, a brown, solid mass remained, which, on treatment with water, reacted violently, liberating a small quantity of liquid isopentane and about 300 c.c. of an inflammable gas. This gas consisted of 59 per cent. of hydrogen, 38 per cent. of isopentane vapour, and about 2 per cent. of unsaturated hydrocarbons.

* The isoamyl halides used in these experiments were obtained from Kahlbaum, and were probably mixtures of several compounds; the results therefore apply to such mixtures and not to pure isoamyl derivatives.—J. F. S.

Interaction of Aluminium and isoAmyl Chloride.

Five grams of isoamyl chloride and 1.2 grams of powdered aluminium were heated for six hours in a sealed tube at 270°. When the tube had cooled, it was immersed in liquid air and opened, the gas being collected. The gas, on analysis, was found to consist of butane, hydrogen, and methane. The solid left was a white mass, which became very hot on treatment with water, and evolved a gas consisting of 96 per cent. of hydrogen, 2 per cent. of isopentane vapour, and small quantities of olefines and acetylenes. There was also a small amount of liquid isopentane.

Interaction of Aluminium and sec-Octyl Iodide.

Five grams of sec.-octyl iodide and 0.5 gram of aluminium were gently heated in a small, hard-glass flask, fitted with a reflux condenser. The reaction commenced after two to three minutes' heating, and rapidly became very violent, hydrogen iodide and iodine being liberated. The product did not solidify on cooling, probably because the intermediate compound was soluble in the excess of octyl iodide. On treatment with water, heat was evolved, and the product, on distillation in a current of steam, yielded 54 per cent. of the theoretical quantity of octane.

Interaction of Aluminium and Iodobenzene.

This reaction was first tried by the ordinary Grignard method. Ten grams of iodobenzene and 1.3 grams of finely-powdered aluminium were placed in a flask containing 50 c.c. of absolute ether; the whole was heated to the boiling point of the ether for two hours without any reaction occurring.

Ten grams of iodobenzene and 2.6 grams of finely-divided aluminium were then gently heated in a small, hard glass flask, fitted with a condenser. After about forty-five minutes, white fumes, consisting chiefly of hydrogen iodide, were evolved, and there was also an odour of benzene, due, no doubt, to the action of atmospheric moisture on the intermediate compound. The reaction took place slowly, and was never violent. After heating for about five hours, the mass was dark brown in colour, and solidified on cooling. The solid was carefully treated with water, when it became very hot and benzene was liberated. The whole was then distilled in a current of steam, and the distillate consisted of small quantities of diphenyl and a 25 per cent. yield of benzene.

Interaction of Aluminium and Chlorobenzene.

Five grams of chlorobenzene and 1.3 grams of finely-powdered aluminium were heated for nine hours in a sealed tube at 270°. The contents of the tube, which were always charred, yet, nevertheless, contained a white, crystalline substance, were treated with water, when much heat was evolved. On distillation in a current of steam, 39 per cent. of the theoretical yield of benzene was obtained.

Interaction of Aluminium and p-Chloroaniline.

Ten grams of *p*-chloroaniline and 2.0 grams of finely-divided aluminium were heated together in a hard glass flask, fitted with a condenser. The reaction proceeded very quietly, and was complete in about four hours. When cold, water was added, and a violent reaction took place with evolution of heat. The whole was then distilled in a current of steam, and 36 per cent. of the theoretical quantity of aniline, together with a small amount of benzidine, was obtained.

Interaction of Aluminium and p-Bromoaniline.

Ten grams of *p*-bromoaniline and 0.8 gram of finely-divided aluminium were heated gently in a hard glass flask. After about five minutes the reaction commenced, and hydrogen bromide was liberated; no further heating was necessary to complete the reaction. When the products were cold, water was added, when a slight evolution of heat occurred. After distilling the products in a current of steam, the distillate yielded 71 per cent. of the theoretical quantity of aniline and a small quantity of benzidine.

Interaction of Aluminium and α -Bromonaphthalene.

This reaction only takes place successfully when the finest aluminium powder is employed; if the somewhat coarser powder, which reacts quite well in other cases, is used, the action is slow and only superficial.

Ten grams of α -bromonaphthalene were mixed in a hard glass flask with 1.4 grams of the finest aluminium powder, and heated for about two to three hours on a sand-bath; for a considerable time the only change observable was a darkening of the α -bromonaphthalene, but suddenly, after the mixture had been heated for about two and a-half hours, a violent reaction set in, hydrogen bromide and naphthalene were evolved and expelled from the top of the condenser, and a general charring of the mixture occurred. The mass was cooled, and then

1832 INTERACTION OF ALUMINIUM, ETC., AND ALKYL HALIDES.

treated with water; there was no evolution of heat, and apparently on reaction took place. The whole was then distilled in a current of steam, and a yield of naphthalene, about 55 per cent. of the theoretical, was obtained. There can be no doubt that a large quantity of the naphthalene was lost both by volatilisation and charring. All attempts to modify the violence of the reaction were without result.

Attempts were made to ascertain if α -bromonaphthalene and aluminium would react in the presence of ether, but these also led to a negative result.

Interaction of Indium and α -Bromonaphthalene.

Five grams of α -bromonaphthalene and 1.4 grams of indium, cut into small pieces, were placed in a silica flask, fitted with an air condenser, and heated. The reaction took place very slowly, and was noticeable at first only by the darkening of the liquid. After heating for about six hours, white crystals appeared in the condenser. The mixture was heated in all for about thirty hours, and then water was added, no heat being evolved. The whole was distilled in a current of steam, when a 45 per cent. yield of naphthalene was obtained.

Interaction of Indium and Iodobenzene.

Five grams of iodobenzene and 2.5 grams of indium were heated for fifteen hours in a sealed tube at 250°. On opening the tube, quantities of hydrogen iodide were evolved. The solid residue, which was dark brown in colour and contained white crystals, was treated with water, when no heat was evolved, but, on distillation in a current of steam, a yield of about 30 per cent. of benzene was obtained.

Interaction of Thallium and α -Bromonaphthalene.

9.8 Grams of thallium, in small pieces, were placed in a flask containing 10 grams of α -bromonaphthalene. This was then heated at the boiling point for twenty-four hours, when a slow reaction occurred, as was evidenced by the formation of a brown deposit on the flask and a white sublimate in the condenser.

When the products had cooled, it was noticed that the thallium was coated with a yellow deposit resembling thallic bromide. The addition of water produced no evolution of heat, but, on distilling in a current of steam, a yield of 17 per cent. of naphthalene, together with a large amount of unchanged α -bromonaphthalene, was obtained. After the naphthalene had been distilled over, there remained in the flask a brown, organic substance containing thallium, but we have been

unable to investigate this substance, since it decomposes on heating, and is insoluble in all the ordinary solvents. Attempts to bring about a reaction between thallium and α -bromonaphthalene in ethereal solution were unsuccessful.

Interaction of Thallium and Iodobenzene.

Five grams of iodobenzene and 5 grams of thallium were heated in a small flask for about twenty hours. The thallium was coated with a red deposit after the heating had been in progress about one and a-half hours, and this red deposit increased as the heating was continued, until finally the thallium had increased in bulk to about three times its original volume. There was no action on the addition of water, but, on distillation in a current of steam, a small yield of diphenyl was obtained. The red compound was thallous iodide, formed probably by the action of the atmospheric moisture on the intermediate compound first produced. Although benzene was not obtained finally, there is no doubt that it was formed and volatilised.

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CLXXXII.—*The Interaction of Hydrogen Dioxide and Sulphides.*

By MAUD GAZDAR and SAMUEL SMILES.

MÄCKER, in 1865 (*Annalen*, 1865, 136, 89), first showed that sulphides may be converted into sulfoxides by nitric acid of a suitable strength, and the process has since been generally applied for this purpose. The yields obtained by the method, however, are often not very good; the aliphatic sulphides are partly destroyed, and the aromatic, in some cases, may be nitrated. One of the present authors, desiring to obtain thionyl diglycollic acid, made several attempts to prepare it from thiodiglycollic acid in the usual manner, but in every experiment it was found that the reagent either was too energetic and completely shattered the molecule of the acid, or, owing to too great dilution, it did not react. The following experiments were therefore undertaken with the view of finding a milder oxidising agent which could be applied to sensitive sulphides, such as the previously-mentioned acid. We have found hydrogen dioxide to be very suitable for this purpose; in acetone or aqueous solution at the ordinary temperature, it converts the sulphide into the sulfoxide almost quantitatively. As may be seen

from the experiments subsequently described, the reagent is successful with simple sulphides of all the chief types, and it may be especially recommended for preparing sulfoxides, which are destroyed or altered by the excess of nitric acid employed in the older method. As examples of the utility of the reagent under these circumstances, the preparation of thionyl diglycollic acid and 4:4'-diaminodiphenyl sulfoxide may be quoted.

These experiments were completed some months ago, and the results are now published, since quite recently Hinsberg has dealt (*Ber.*, 1908, 41, 2836) with the action of hydrogen dioxide on organic sulphur derivatives, but more particularly with the aromatic disulphides. Hinsberg observes that the sulfoxides are not converted into the sulphones by excess of hydrogen peroxide at the ordinary temperature; we have also studied this question, and concur fully in this conclusion with regard to the substances here dealt with.

Diisooamyl Sulphoxide, (C₅H₁₁)₂SO.

Five grams of diisooamyl sulphide were mixed in acetone solution with a slight excess of concentrated (30 per cent.) aqueous hydrogen peroxide. The solution was set aside at the ordinary temperature for twenty-four hours, and then the acetone was evaporated and the residue recrystallised from light petroleum. The yield of the purified product, which melted at 35°, was 3 grams:

0.1063 gave 0.2450 CO₂ and 0.1109 H₂O. C = 62.8; H = 11.6.

C₁₀H₂₂OS requires C = 63.16; H = 11.58 per cent.

Saytzeff (*Annalen*, 1866, 139, 354) described this sulfoxide as melting at 37–38°, and, since the sulphone melts at 31°, we prepared a fresh specimen of amyl sulfoxide by the nitric acid method. The product melted at 35°, and a mixture of sulfoxides from the two sources melted at the same temperature.

Thionyl diglycollic Acid, SO(CH₂·CO₂H)₂.

A solution of thiodiglycollic acid in excess of aqueous hydrogen peroxide was set aside for forty-eight hours at the ordinary temperature. The excess of water was then evaporated, and the residue, which was at first a syrup, became solid on being kept under diminished pressure over sulphuric acid. The product, which melted at 79–80°, was evidently pure:

0.1618 gave 0.1728 CO₂ and 0.0589 H₂O. C = 29.1; H = 4.0.

C₄H₆O₅S requires C = 28.92; H = 3.61 per cent.

The barium salt was prepared by the action of barium carbonate on the aqueous solution of the acid. It is sparingly soluble in water, and

separates from the hot medium in minute crystals containing two molecules of the solvent to one of the salt :

0.2604, heated to 130° , lost $0.0276 \text{ H}_2\text{O}$. $\text{H}_2\text{O} = 10.6$.

0.2180 " " " $0.0228 \text{ H}_2\text{O}$. $\text{H}_2\text{O} = 10.46$.

0.2869 gave 0.1989 BaSO_4 . $\text{Ba} = 40.6$.

$\text{C}_4\text{H}_4\text{O}_6\text{SBa} \cdot 2\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 10.68$; $\text{Ba} = 40.54$ per cent.

The lead salt was obtained as a white, curdy precipitate on mixing aqueous solutions of the acid and lead acetate; it is insoluble in boiling water, but is slightly decomposed thereby. The air-dried substance was analysed :

0.2315 gave 0.189 PbSO_4 . $\text{Pb} = 55.8$.

$\text{C}_4\text{H}_4\text{O}_6\text{SPb}$ requires $\text{Pb} = 55.8$ per cent.

The gelatinous silver salt is insoluble in water, and, when dried, forms a yellow, horny mass. It is scarcely acted on by boiling methyl iodide.

Dibenzyl Sulphoxide, $(\text{C}_6\text{H}_5 \cdot \text{CH}_2)_2\text{SO}$.

This was prepared by oxidation of dibenzyl sulphide in acetone solution with the calculated quantity of hydrogen peroxide. The yield was quantitative, and the recrystallised product melted at 134° , whether alone or mixed with a sample of the sulphoxide prepared in another manner. Excess of the oxidising agent also gave the sulphoxide.

4:4'-Diethoxydiphenyl Sulphoxide, $(\text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{C}_2\text{H}_5)_2\text{SO}$.

4:4'-Diethoxydiphenyl sulphide was oxidised in acetone solution with excess of aqueous hydrogen dioxide. The yield was almost theoretical, and the product melted at 116° and gave the characteristic Prussian-blue colour with concentrated sulphuric acid, which was discharged by addition of phenetole :

0.1682 gave 0.1395 BaSO_4 . $\text{S} = 11.4$.

$\text{C}_{16}\text{H}_{18}\text{O}_6\text{S}$ requires $\text{S} = 11.03$ per cent.

4:4'-Diaminodiphenyl Sulphoxide, $(\text{C}_6\text{H}_4 \cdot \text{NH}_2)_2\text{SO}$.

Diaminodiphenyl sulphide was treated in acetone solution at the ordinary temperature with the calculated quantity of hydrogen dioxide. After twenty-four hours had elapsed, the acetone was evaporated, and the residue, which was at first a syrup, crystallised when set aside for some hours in a desiccator under diminished pressure. The slightly coloured product was purified with animal charcoal in alcoholic or acid aqueous solution. 4:4'-Diaminodiphenyl sulphoxide was thus obtained in colourless prisms, which melted with decomposition at 175° . The substance gives with concentrated

sulphuric acid a deep bluish-green colour, which is discharged by phenetole:

0.6908 gave 0.2049 CO_2 and 0.0395 H_2O . $\text{C} = 61.5$; $\text{H} = 4.8$.

$\text{C}_{12}\text{H}_{12}\text{ON}_2\text{S}$ requires $\text{C} = 62.0$; $\text{H} = 5.1$ per cent.

Tetramethyldiaminodiphenyl sulphide was treated in the same manner, and a substance melting at 135° was obtained, but this was not further investigated, since, not giving the characteristic reaction with sulphuric acid, it did not appear to be a sulphoxide. It may be mentioned that diphenylene disulphide and tetranitrodiphenyl disulphide are not attacked by hydrogen dioxide under the conditions described in this paper.

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CLXXXIII.—*The Influence of Solvents on the Rotation of Optically Active Compounds. Part XIII.* Ethyl Tartrate in Aromatic Nitro-derivatives. Influence of Temperature-change on Rotation in Solution.*

By THOMAS STEWART PATTERSON.

IN former parts of this investigation, it has been shown that the specific rotation of ethyl tartrate may vary to a very considerable extent according to the nature of an inactive substance in which it may be dissolved. Water, it was found, at $20^\circ\text{C}.$ † and infinite dilution, raised the rotation to $+26.8^\circ$ (Trans., 1904, 85, 1130), whilst chloroform depressed it to -3.0° (Trans.,[‡] 1905, 87, 313; 1908, 93, 357). Later on, Winther observed that in formamide the rotation attains to a somewhat higher value than in water, namely, $+30.4^\circ$ (Zeitsch. physikal. Chem., 1907, 60, 578), whilst ethylene bromide produces a lower rotation (-19°) than chloroform (*ibid.*, p. 578). Finally, in the previous part of this series of papers it was shown that acetylene tetrabromide has a still more powerful depressing effect than ethylene bromide, reducing the rotation of the active ester to -20° (Trans., 1908, 93, 360). Thus the range through which the rotation may vary, from $+30.4^\circ$ to -20° , is great, at least when

* For Part XII, see Trans., 1908, 93, 936. All experimental details, meaning of terms, &c., are as previously given.

† Degrees of temperature are indicated in this manner throughout this paper to avoid confusion with degrees of rotation.

compared with the rotation at 20°C. (+7.85) of the ester itself. In the following pages, however, it will be shown that both the upper and lower value may be very considerably exceeded. On this account, and also because of the modifications of rotation caused by change of temperature, the present communication may perhaps claim to be of some interest, not only in regard to the problems of optical rotation, but to the more general question of solution as well.

The influence of a solvent is, until we find reason for a more suitable plan, best ascertained by comparison of rotation values for one definite temperature. Therefore, from the curves obtained by examining the rotation of a number of solutions at various temperatures, values at 20°C. have been obtained by interpolation or extrapolation, as the case may be. Complete and trustworthy concentration curves, however, can only be drawn for such solvents as are either liquid, or else have a comparatively low melting point, and of the solvents dealt with here only four conform to these conditions. The curves obtained for the rotation of ethyl tartrate in these four solvents are shown in Fig. 1, which gives a general idea of the influence of this class of solvents, and illustrates in particular the manner in which solvent influence varies with concentration.

It will be observed that all four solvents have powerful and similar effects. In each case the rotation of the dissolved ester is markedly raised, and, as the dilution becomes greater, the rotation increases at a gradually increasing rate.

The specific rotation of pure ethyl tartrate is +7.85°. In nitrobenzene solutions, the interpolated values at 20°C. are as follows:

p	50.02	19.94	10.0	5	2.002
$[\alpha]_D^{20}$	17.0°	26.1°	31.6°	34.9°	38.4°

By extrapolation, the specific rotation at infinite dilution would be +41°.

The general behaviour of *o*-nitrotoluene is very much the same as that of nitrobenzene, the data being:

p	50.21	25.01	10.002	5.002
$[\alpha]_D^{20}$	17.8°	25.0°	32.3°	36.5°

At each dilution examined, the rotation is approximately one degree greater than in nitrobenzene, so that the value at infinite dilution is very nearly +42°.

In *m*-nitrotoluene, the data are:

p	51.2	25.0	9.99	5.0
$[\alpha]_D^{20}$	13.0°	18.2°	+25.0°	28.7°

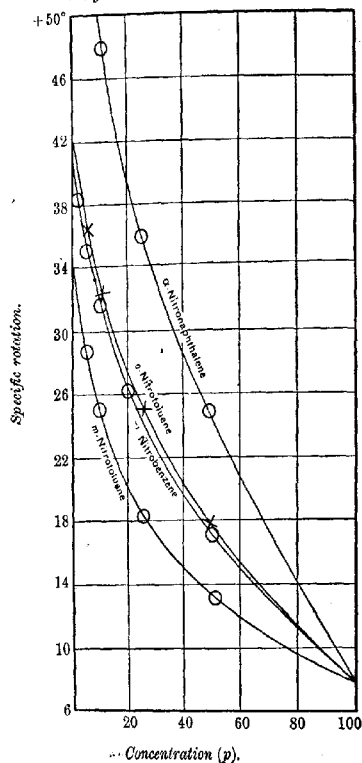
The effect in this case is very considerably less than in the two previous solvents. At infinite dilution, the rotation is +34°.

In *o*-nitronaphthalene, the greatest solvent influence of all is found, the data being :

p	49.56	25.14	10.214
$(a_p)^{25}$	25.0°	35.9°	47.9°

FIG. 1.

Relationship of specific rotation and concentration.
Ethyl tartrate in various solvents.



The increase of rotation in dilute solution on further addition of solvent becomes very rapid indeed, so that at infinite dilution the rotation of the dissolved ethyl tartrate would be about +65°, or more than twice as great as in formamide.

As has already been mentioned, the data for the other solvents examined are not so complete. These substances being solids, sometimes of fairly high melting point, they were naturally more difficult to work with, and fewer solutions were examined. Since estimates of the value of the specific rotation at infinite dilution in these solvents can scarcely be made, it will be necessary to compare data for somewhat concentrated solutions, about $p = 25$ in most cases, and it must be remembered that even then the numbers used are obtained by extrapolation. A $p = 25$ solution in *s*-trinitrobenzene, for instance solidifies at about 100°C ., so that the specific rotation quoted below for 20°C . can obviously only be an approximation. In such cases, the rotation observations can be made easily enough, but the density determinations present greater difficulty, and in some instances were omitted altogether. Between two observations, the solvent crystallises out, and the re-melting of it is attended by the disadvantage that some of the ethyl tartrate, which along with a portion of the solvent remains liquid, is apt to be expelled from the tube in greater proportion than the solvent. The concentration of the solution may thus become altered, and make the density determinations untrustworthy.

Specific rotations cannot therefore be calculated for every solution examined, so, in order to obtain numbers directly comparable for all the solvents, the observations have been reduced to a common standard by means of the usual specific rotation formula, but omitting the density.

For convenience of reference, we may assign to this expression the letter R .

$$\text{Thus:} \quad R_D^c = \frac{a_D^c \times 100}{l \times p}$$

The Relationship between Constitution and Solvent Effect.

Only the narrower aspect of this problem can meantime be discussed, and the material will be found in the table on p. 1840.

From these data, some generalisations can be made. In the first place, it may be noted that *o*-nitrotoluene causes a slightly greater rise in the rotation of ethyl tartrate than does nitrobenzene. The difference, however, in the values of R_D^{20} for $p = 25$ solutions is very slight—only 0.2° . The methyl group in the ortho-position, then, enhances, but only by a small amount, the effect of the nitro-group. The introduction of the methyl group in the meta-position is accompanied by a considerable change—a depression in this case—the rotation in *m*-nitrotoluene ($R_D^{20} = 21.2^{\circ}$) being, roughly, 9° lower than in nitrobenzene. The change in position of the methyl group from the meta-

TABLE I.

Rotation of Ethyl Tartrate.

Solvent.	[α] _D ^{20°}		R _D ^{20°}
	p=0.	p=25.	p=25.
Nitrobenzene	+41*	+24.2°	+29.1°
<i>o</i> -Nitrotoluene.....	42	25.0	29.3
<i>m</i> -Nitrotoluene ..	34	18.2	21.2
<i>p</i> -Nitrotoluene	28*	17.2	20.1
<i>o</i> -Dinitrobenzene.....	—	36.0*	46.0
<i>m</i> -Dinitrobenzene †	—	18.0	25.3
2:6-Dinitrotoluene	—	23.3	31.5
2:4-Dinitrotoluene	—	11.7	15.5
<i>s</i> -Trinitrobenzene	-30*	-22.0*	-30.0
α -Nitronaphthalene †.....	+65	+35.9	+44.5

* These numbers must be regarded as rough approximations only.

† It was intended also to examine *p*-dinitrobenzene and β -nitronaphthalene, but these substances are so dark in colour that experiments similar to those the results of which are recorded in the table could not be carried out.

It was found possible to compare the influence of β -nitronaphthalene with that of α -nitronaphthalene (see p. 1842), but the rotation of a solution containing only 3.63 per cent. of *p*-dinitrobenzene could not be determined with a sufficient degree of accuracy in a 40 mm. tube.

to the para-position, on the other hand, produces a much less effect; the rotation of ethyl tartrate in *p*-nitrotoluene ($R_D^{20} = 20.1^\circ$) shows a further diminution of just over a degree.

These examples appear to show that the relative positions of the substituent groups play an important part in determining the rotation of the dissolved ester. The only data from previous work which can be cited in comparison are those relative to *o*-, *m*-, and *p*-xylene (Trans., 1902, 81, 1107), which are as follows:

Ethyl tartrate in	Benzene.	Toluene.	<i>o</i> -Xylene.	<i>m</i> -Xylene.	<i>p</i> -Xylene.
[α] _D ^{20°} (infinite solution)	+6.1°	4.8°	2.7°	1.8°	0.7°

The enormous effect of the introduction of the nitro-group into benzene is most striking, the rotation of the dissolved ethyl tartrate being raised from [α]_D^{20°} (inf. dil.) = 6.1° in benzene to [α]_D^{20°} (inf. dil.) = 41° in nitrobenzene. The introduction of the nitro-group in the three possible positions in toluene also brings about a great increase in rotation with the gradation which has just been described; in the ortho-position the two groups assist one another, in the meta-position they detract from each other's influence, and still more so in the para-

position. It will be seen that in *o*-, *m*-, and *p*-xylenes the phenomena are at least of an analogous character. One methyl group introduced into benzene exercises a depressing influence; two methyl-groups, even in the ortho-position, have a greater depressive effect, which becomes still more marked for *m*- and *p*-xylene. In both series, the rotation of the active ester is greatest when the two substituting groups are in the ortho-position, and least when they are in the para-position.

The first of the dinitro-derivatives to be examined was *m*-dinitrobenzene. Since one nitro-group has so great an influence, it might have been expected that a second such group anywhere in the molecule would augment the solvent effect to a still greater degree. Just the opposite is the case, however. In *m*-dinitrobenzene, the value $R_D^{20} = 25.8^\circ$ is considerably less than for nitrobenzene (29.1°), so that the elevating tendency of the groups is largely overcome by a secondary cause, namely, their influence on one another, due in turn to their relative position in the molecule.

The inference which could be drawn from the behaviour of the nitrotoluenes as to the mutual influence of two substituting groups led to the expectation that *o*-dinitrobenzene would have at least a greater effect than *m*-dinitrobenzene. This was found to be very markedly the case; in *o*-dinitrobenzene the value $R_D^{20} = +46^\circ$ is much greater even than in nitrobenzene. Thus the influence of one substituting nitro-group on another is like the effect of a methyl group on a nitro-group, but very much more pronounced; *o*-dinitrobenzene ($R_D^{20} = 46^\circ$) causes a much higher rotation than *o*-nitrotoluene ($R_D^{20} = 29.3^\circ$); *m*-dinitrobenzene ($R_D^{20} = 25.8^\circ$) causes a higher rotation than *m*-nitrotoluene (21.2°).

The two dinitrotoluenes examined display just the same regularities. Both these compounds are substituted *m*-dinitrobenzenes. In 2:6-dinitrotoluene, the methyl group is in the ortho-position with respect to both nitro-groups, and might be expected to enhance their influence—perhaps by about twice as much as is the case in *o*-nitrotoluene. The increase is somewhat greater than this, the rotation in 2:6-dinitrotoluene ($R_D^{20} = 31.5^\circ$) exceeding that in *m*-dinitrobenzene ($R_D^{20} = 25.8^\circ$) by about five degrees.

In 2:4-dinitrotoluene, the methyl group is ortho with respect to one nitro-group and para with respect to the other. By comparing *o*-nitrotoluene and *p*-nitrotoluene respectively with nitrobenzene, the former might be expected to raise the rotation slightly, about 0.2° , and the latter to diminish it by about 9° . The total change should therefore be a diminution of about 9° . The data are 2:4-dinitrotoluene, $R_D^{20} = 15.5^\circ$, *m*-dinitrobenzene, $R_D^{20} = 25.8^\circ$, so that the lowering of rotation due to the methyl group is 10.3° .

Whilst an agreement so close as this is probably a coincidence, it appears, nevertheless, that in a qualitative sense the variation of solvent influence with change of relative position of the substituent groups can be traced with some success.

After the experience gained with the foregoing solvents, it was to be expected that *s*-trinitrobenzene (m. p. 123°) would cause a lower rotation than *m*-dinitrobenzene, but the actual behaviour could scarcely have been foreseen. Not only is the rotation of the dissolved ethyl tartrate lower than in *m*-dinitrobenzene, or for the homogeneous ester, but it is lowered to such an extent that it has a considerable negative value. The concentration-rotation curve has therefore an appearance quite of the opposite character to those of Fig. 1. The rotation of the ester diminishes rapidly as *s*-trinitrobenzene is added, so that, at 20°C., inactivity would be reached even in a solution so concentrated, with reference to ethyl tartrate, as $p=82$. At $p=50$, the rotation would be approximately $R_D^{20} = -15^\circ$; at $p=25$, about -30° , and at infinite dilution, about -47.5° . The density of trinitrobenzene in the liquid form at 20°C. would be about 1.606*, whence the specific rotation at infinite dilution would be approximately $[\alpha]_D^{20} = -30^\circ$, a value considerably below that hitherto recorded for ethyl tartrate in any other solvent.†

It has been pointed out already that α -nitronaphthalene has a very powerful influence on the rotation of ethyl tartrate, the specific rotation at infinite dilution reaching the value $+65^\circ$.‡ By the use therefore of α -nitronaphthalene, on the one hand, and *s*-trinitrobenzene, on the other, the rotation of the ethyl tartrate may be varied at will almost through 100° , which must be regarded as a very considerable change indeed.

The relative influences of α - and β -nitronaphthalene were determined by comparison of the rotations, determined in a short tube, of solutions containing a large proportion of ethyl tartrate.

For α -nitronaphthalene ($p=93.94$), the data were α_D^{20} (40 mm.) $= +4.68^\circ$, whence $R_D^{20} = +12.46^\circ$, whereas for β -nitronaphthalene ($p=92.72$), α_D^{20} (40 mm.) $= +4.09^\circ$, whence $R_D^{20} = +11.02^\circ$.

Thus, even in solutions so dilute with reference to the inactive component as these, α -nitronaphthalene causes a value for R_D^{20} practically 1.5° greater than does the β -isomeride. It need scarcely be pointed out that this is entirely in harmony with the other regu-

* This value was estimated from two determinations made for the purpose. At 137°C., $d_{20/4} = 1.4863$, and at 170°C., $d_{20/4} = 1.4523$.

† The behaviour of *s*-trinitrobenzene is thus in agreement with that of mesitylene (Trans., 1902, 81, 1099), which latter solvent also causes a negative rotation in dilute solutions of ethyl tartrate.

‡ This high rotation would probably be equalled, or nearly equalled, in *o*-dinitrobenzene.

larities discussed, since α -nitronaphthalene may be regarded as an ortho- and β -nitronaphthalene as a meta-substitution product.

It will be seen from the table below that just as nitrobenzene has a much greater solvent influence than benzene, so α -nitronaphthalene has a much greater influence than naphthalene. Also, that whilst naphthalene has a greater influence than benzene, so naphthalene derivatives have greater influences than benzene derivatives.

TABLE II.

Rotation of Ethyl Tartrate.

Solvent.	$[\alpha]_D^{20}$ (infinite dilution).	Solvent.	$[\alpha]_D^{20}$ (infinite dilution).	Δ .
Naphthalene *	+32°	Benzene †	+6.1°	25.9
α -Bromonaphthalene †	26	Bromobenzene †	11.7	14.3
α -Nitronaphthalene	65	Nitrobenzene ...	41.0	24.0

* Trans., 1902, 81, 1134. † Trans., 1903, 93, 936. ‡ Trans., 1902, 81, 1097.

The sequence, however, is not the same in the two series, α -bromonaphthalene causing a lower rotation than naphthalene, whilst bromobenzene brings about a considerably higher rotation than benzene.

Influence of Temperature-change on Rotation in Solution.

Whereas no prediction can meanwhile be made as to the probable effect of a solvent, of a type not already investigated, on the rotation of ethyl tartrate, the influence of temperature-change on such solutions seems to be more simple in character. With our previous knowledge, it would have been impossible to foretell that nitrobenzene would have a very elevating influence, or, even knowing the influence of nitrobenzene, that *s*-trinitrobenzene would have a depressing effect. But, knowing the rotations in these two solvents at 20°C., former work allowed of a prediction to be made as to the effect of temperature-change. Since the rotation in nitrobenzene is much above that of the homogeneous ester, it was to be expected that, with rise of temperature, the rotation would diminish, whilst in *s*-trinitrobenzene, in which the specific rotation is far below that of the undiluted ester, rise of temperature should bring about a rapid increase of rotation, and this is, indeed, decidedly the case.

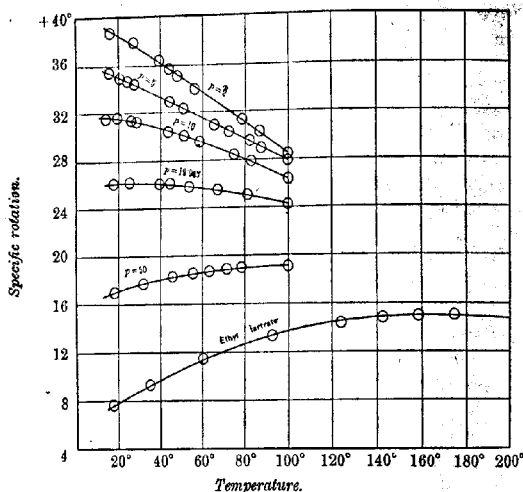
Figures 2 and 3 will help to elucidate the very interesting effects of temperature-change on the rotation of these solutions. In the former are shown temperature-rotation curves for ethyl tartrate in nitrobenzene along with the curve for the ester alone.*

* A number of the solutions dealt with in this paper were examined before the author was in possession of an apparatus that allowed of determinations above 100°C.

The curve for ethyl tartrate (Fig. 2) has a maximum specific rotation (+15°) at 175°C.* The appearance of the curve for a $p=50$ solution would lead us to expect a maximum at about 105°C, at a lower temperature therefore, but having a higher value ($[\alpha]_D^{25} = 19^\circ$), than in the pure ester. In a $p=20$ solution, the maximum lies at about 30°C, and has the value $[\alpha]_D^{25} = 26.2^\circ$. In a $p=10$ solution, the maximum rotation has a still greater value ($[\alpha]_D^{25} = 31.7^\circ$), and occurs at a lower temperature, 17°C. At less concentrations, the maximum

FIG. 2.

Relationship between rotation and temperature.
Ethyl tartrate in nitrobenzene.



value lies below the range of temperature investigated, but, as the specific rotation increases with greater dilution, the diminution of rotation with rise of temperature becomes, as in the $p=5$ and $p=2$ solutions, gradually more rapid.

* In order to determine this point, ethyl tartrate was re-examined, with the results recorded on p. 1852. It will be noticed from them that the observed rotation reaches a distinct maximum of +15.93° at 148°C., two observations made at higher temperatures being lower than this. Since, however, the density diminishes with rise of temperature, the maximum specific rotation (+15°) occurs at a higher temperature, 175°C.

Walden (*Ber.*, 1905, 38, 366) gives some data for the rotation of ethyl tartrate at temperatures up to 150° for red, green, blue, and violet light.

This behaviour is of exactly the same character, but perhaps not quite so obvious as that of methyl potassium tartrate, ethyl potassium tartrate, and *n*-propyl potassium tartrate in aqueous solution (Trans., 1904, 85, 1121, 1123, 1125). For each of these three substances a very distinct maximum is observable at temperatures between 100°C. and 20°C. in solutions of concentration below about $p=50$. As the dilution becomes greater, these maxima increase in value, and move to a lower temperature, the relationship between the value of the maximum and the temperature at which it occurs being apparently linear (*loc. cit.*, p. 1137). The whole temperature range of the maxima is, however, comparatively slight, varying, for instance, from 56°C. for a $p=48.79$ solution of ethyl potassium tartrate to 38°C. for a $p=5.07$ solution.

For ethyl tartrate in nitrobenzene, the temperature at which this maximum rotation occurs varies through very much wider limits, passing from 175°C. for $p=100$ to 17°C. for $p=10$.

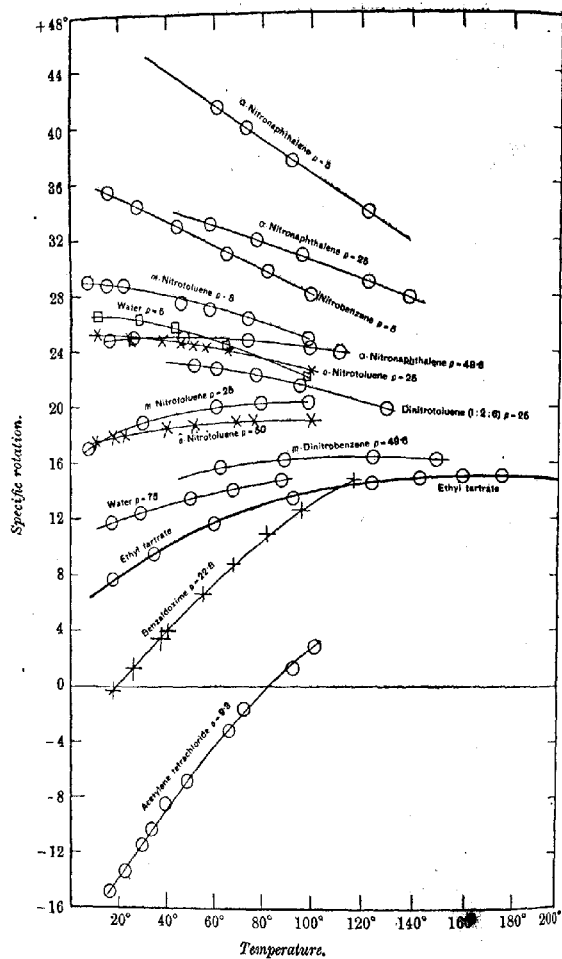
In the three alkyl potassium tartrates, the maximum rotations lie along a different line for each substance, which, of course, is only to be expected. An interesting question at once suggests itself as to the variation of the maximum rotation for one and the same active substance in different solvents. The maxima must start from one point, the maximum rotation of the pure ester, in all solvents; will they follow the same course or not?

For the discussion of this question, it is unnecessary to reproduce all the curves for each solvent—they exhibit the same characteristics as those for nitrobenzene—but in Fig. 3 are shown temperature-rotation curves for ethyl tartrate in different solvents, and at various concentrations, which present some interesting features.

The curve for ethyl tartrate is included as before, the maximum specific rotation ($+15^\circ$) lying about 175°C. The next curve above it is for water, $p=75$; it extends only to 100°C., but from the shape of the curve a maximum would probably occur at a lower temperature than in the homogeneous ester. The next curve, for *m*-dinitrobenzene, $p=49.611$, shows a maximum ($[\alpha]_D^{25}=16.2^\circ$) at 123°C. The curve for *o*-nitrotoluene, $p=50.21$, extends also only to 100°C., but its appearance indicates a maximum ($[\alpha]_D=18.8^\circ$) about that temperature. The next curve represents both *m*-nitrotoluene, $p=25.003$, and *p*-nitrotoluene, $p=20$, the two curves being indistinguishable from each other. The maximum ($[\alpha]_D=20.2^\circ$) occurs at 85°C. From the appearance of the other curves, it is clear that the maximum passes rapidly to lower temperatures as the rotation increases.* In α -nitronaphthalene, $p=49.565$, $\frac{1}{2}$ has the value $[\alpha]_D=24.9^\circ$ at 42°C.; in nitrobenzene, $p=10$, its value is about $[\alpha]_D=31.7^\circ$, and it lies at 17°C.

* This behaviour is, to some extent, reminiscent of the passage of the equilibrium between water and ice to a lower temperature with increase of pressure.

FIG. 3.
Influence of temperature on rotation in solution.
Ethyl tartrate in various solvents.



At lower temperatures, it is obviously more difficult to determine the positions of the maximum. It seems, however, that the temperature at which a maximum rotation of given value occurs does not vary much in different solvents.*

There are also included in Fig. 3 some curves for solvents which have a powerful depressing influence on the rotation of ethyl tartrate, and it will be seen that their appearance for the most part is in harmony with the thesis just developed. When the rotation at 20°C. is very low, as in acetylene tetrachloride or *s*-trinitrobenzene, it increases very rapidly on heating, and, although the point has not been experimentally proved, the maximum rotation in such solvents may lie above 175°C.

A curve of this character for benzaldoxime has also been included in the figure, for the purpose of showing that exceptions have to be reckoned with. Although the rotation is much depressed at low temperatures, this curve tends to cut that for the homogeneous ester, and is therefore not in agreement with the foregoing statement. Such exceptions are, however, so far as can be judged at present, quite rare.

It will be clear, then, that Fig. 3 bears a striking resemblance to Fig. 2 in general appearance; the curves for these various solvents taken in this indiscriminate manner blend into a diagram remarkably similar to that for one single solvent at a number of concentrations, and it would seem as if we might almost eliminate from consideration the nature of the inactive solvent and the concentration. The course

* Winther (*Zeitsch. physikal. Chem.*, 1903, 55, 347) refers to this subject of maximum rotation in regard to solutions of ethyl tartrate in *isobutyl* alcohol. By means of interpolation formulae, he calculates the temperature (T) at which the maximum rotation would occur for different concentrations, and finds, for example, the following numbers. (It should be noticed that the numbers in the third line are not the values of the maximum rotation.)

p	80.97	62.56	41.24	21.61	13.82
T	140°C.	140°C.	100°C.	118°C.	141°C.
$[\alpha]_D^{20}$...	5.78°	5.18°	4.98°	5.31°	5.42°

Winther's conclusion from these and other similar figures is that the temperature of maximum rotation of ethyl tartrate in *isobutyl* alcohol does not vary with the concentration, and this is in agreement with the present results. It is only in a very powerful solvent like nitrobenzene that the maximum is displaced sufficiently for the fact to be noticeable. Fig. 3 shows that even when rotations are very different at 20°C., as, for instance, in nitrobenzene, $p=5$, and in homogeneous ethyl tartrate, they are much closer at 100°C. If they lie close together at 20°C., as is the case in *isobutyl* alcohol, there will certainly be very little difference between them at high temperatures. The above values for T are considerably too low, as is also the maximum temperature (137°C.) suggested by Winther for pure diethyl tartrate [*loc. cit.*, p. 347].

of the temperature-rotation curve is apparently defined by the rotation at any given temperature.*

If, as a first approximation, we assume this to be so, it may be possible to find an expression by means of which the rotation of ethyl tartrate at one temperature, say, 100°C., can be calculated, given that at another temperature, say, 20°C., without regard to the solvent or the concentration.

It is clear from Figs. 2 and 3 that, in such solvents as have a powerful effect in raising the rotation of ethyl tartrate, there must generally, or always, be some solution for which the rotation has the same value at 20°C. and at 100°C., that is, one in which the maximum rotation falls between 20°C. and 100°C. Let this rotation be X° , then if the rotation of a solution be greater than X° at 20°C. it will diminish on heating, and vice versa. Therefore we may write

$$k(X^\circ - [\alpha]_D^{20}) = [\alpha]_D^{100} - [\alpha]_D^{20} \dots (1)$$

as the simplest kind of expression connecting the rotations at 20°C. and 100°C.

The values of the constants k and X° may then be determined from the rotation data for any two solutions. Thus, substituting the values of $[\alpha]_D^{100}$ and $[\alpha]_D^{20}$ for solutions in nitrobenzene, of $p=5$ and $p=50$ (see table below), we find $X^\circ = +20.96^\circ$ and $k=0.505$. Thus the solution (its concentration does not concern us) the specific rotation of which is 20.96° at 20°C., should have the same rotation also at 100°C. It will be apparent from the curves in Fig. 2 that this must be almost correct.

Again, the values of k and X° may be determined from the rotation data for ethyl tartrate in other solvents, and, if the simple assumption made above were correct, should be the same as those just found for nitrobenzene.

In table III are given data for various solvents and concentrations,

TABLE III.

Number.	Solvent.	p .	$[\alpha]_D^{20}$.	$[\alpha]_D^{100}$.
1.	Nitrobenzene	5	+35.0*	+27.9*
2.	"	50	17.0	19.0
3.	<i>o</i> -Nitrotoluene	10	32.3	25.5
4.	Water*	5	26.5	21.9
5.	"	75	11.9	14.6
6.	α -Nitronaphthalene	10	46.0	36.5
7.	Benzaldoxime†	22.8	0.2	12.8
8.	Ethyl tartrate	100	7.8	13.6
	(homogeneous ester)			

* Trans., 1904, 85, 1151.

† Trans., 1907, 91, 506.

* This is, however, not strictly true. It will be observed that the curve for α -nitronaphthalene, $p=25.145$, has not the same inclination as that for nitrobenzene, $p=4.999$; the two curves, in fact, would intersect, but, on the whole, they show remarkable similarity.

by means of which k and X° may be calculated, and in table IV are given values of these two constants eliminated between different pairs.

TABLE IV.

Eliminated between...	1.2.	1.5.	1.8.	1.7.	4.8.	3.5.	6.7.
k	0.505	0.418	0.473	0.565	0.555	0.465	0.483
X°	20.96°	18.36°	20.0°	22.5°	18.21°	17.7°	26.31°

It will be seen that the constants calculated by means of this linear expression from data for two solutions in the same solvent (1.2), or between those for solutions in two different solvents (1.5, 1.7, &c.), or between those for a solution and the homogeneous ester (1.8, 4.8), all give values for the constants lying fairly close together.

Again, these constants, once obtained, may be used for calculating the rotations of other solutions, and, from table V, values calculated in this way may be compared with experimental numbers. The constants used are those first mentioned, and derived from nitrobenzene solutions of $p = 5$ and $p = 50$.

Equation (1) then transforms into :

$$[\alpha]_D^{100} = 0.495 [\alpha]_D^{50} + 10.583^\circ \dots \dots (2).$$

Substituting in this expression the values of $[\alpha]_D^{50}$ of column 4 of table V, the values in column 6 for $[\alpha]_D^{100}$ are obtained.

TABLE V.

Number.	Solvent.	p .	$[\alpha]_D^{50}$.	$[\alpha]_D^{100}$ (observed).	$[\alpha]_D^{100}$ (calculated).
1.	Nitrobenzene	2.0	38.4°	28.5°	29.6°
2.	"	10.0	31.6	26.2	26.22
3.	"	19.94	26.1	24.3	23.5
4.	Ethyl tartrate	100.0	7.85	13.6	14.4
	(homogeneous ester)				
5.	α -Nitronaphthalene.....	5.0	46.0	36.5	33.4
6.	"	49.56	24.8	24.0	22.9
7.	Benzaldoxime *	22.8	0.2	12.8	10.5
8.	"	49.6	10.8	16.1	15.28
9.	"	79.9	12.9	16.4	16.97
10.	m -Nitrotoluene.....	5.0	28.7	24.8	24.79
11.	m -Dinitrobenzene.....	25.0	23.4	21.4	22.2
12.	o -Nitrotoluene	50.0	17.9	18.9	19.4
13.	Mesitylene †	10.0	1.8	11.06	11.5
14.	Acetylene tetrachloride ‡	9.3	-13.8	2.4	3.75

* Trans., 1907, 91, 506. † Trans., 1902, 81, 1129. ‡ Trans., 1908, 93, 366.

Thus the constants calculated from the $p = 5$ and $p = 50$ nitrobenzene solutions give fairly satisfactory results, not only when applied to nitrobenzene solutions of different concentrations (1-3), but when applied to the homogeneous ester itself (4), or to solutions in other solvents so very different in character as α -nitronaphthalene, benzaldoxime, mesitylene, and acetylene tetrachloride (5-13). It seems

particularly worthy of note that a formula thus obtained from rotations varying from $+35^{\circ}$ to $+17^{\circ}$ (1 and 2, table V) should be directly applicable to a solution in acetylene tetrachloride, the rotation of which varies between -13.8° and $+2.4^{\circ}$ (14, table V).

The simplest interpretation of the results of this investigation seems to be one purely physical: either that suggested some eight years ago in the first paper of the series, correlating the phenomena of rotation in solution with internal pressure (Trans., 1901, 79, 188), or one similar in character. It is difficult to believe, considering what we know of actual compounds and the almost complete lack of success which has attended the attempt to discover regularities amongst their rotation constants, that the present results can be due to combination between solvent and solute, although there is one respect in which the phenomena might be held to harmonise with that supposition. There is the fact, namely, that the rotations which are so different at $20^{\circ}\text{C}.$ obviously tend towards a common value at higher temperatures, a behaviour which might, of course, be ascribed to the gradual dissociation of complexes existing at low temperatures. But this does not seem to explain the fact that the maximum rotation of the homogeneous substance is found quite clearly in solution also, or that a maximum rotation of given value occurs at the same, or very nearly the same, temperature in totally different solvents, in other words, that this maximum is related to the temperature at which it occurs in a manner which is, to a considerable extent, independent of solvent and concentration.

Again, it might be expected, at least as a first approximation, that, as an active substance is gradually diluted with an inactive solvent at a given temperature, the combination of the two substances should follow the law of mass action, and that this, in turn, should be reflected in the variation of rotation. The form of the concentration curves in Fig. 1, however, is entirely opposed to this suggestion, since the specific rotation alters more rapidly in dilute than in concentrated solution.*

The suggestion, referred to above, that rotation may be traced back to internal pressure, or, in turn, to solution-volume, can, unfortunately, not be completely tested in the present case, since only three of the solvents dealt with are liquids.

The data for these, however, are given on p. 1856. It appears that at infinite dilution in nitrobenzene the molecular solution-volume of ethyl tartrate would be about 174 c.c., in *o*-nitrotoluene about 175.5 c.c., and in *m*-nitrotoluene about 176.5 c.c. In nitrobenzene,

* See Trans., 1907, 91, 1844, where this question is discussed in regard to other solvents.

ethyl tartrate has thus a smaller volume and a greater rotation than in *m*-nitrotoluene, but in *o*-nitrotoluene the rotation is greater than in either of the other two solvents, and therefore the volume might be expected to be less, which is, however, not the case. The data are thus not in harmony, and it would be of little service in the meantime to do more than cite them.

In conclusion, the results of the present investigation may be summarised as follows :

(1) The rotation of ethyl tartrate varies through very wide limits when dissolved in the aromatic nitro-derivatives. In nitrobenzene and *o*-nitrotoluene, the specific rotation at infinite dilution has a value of more than $+40^\circ$; in α -nitronaphthalene and *o*-dinitrobenzene, it would reach a value of, approximately, $+65^\circ$. In *s*-trinitrobenzene, on the other hand, the specific rotation would lie about -30° , so that the total change of rotation which can be produced by varying the solvent is practically 100° .

(2) The solvent influence of disubstituted benzenes appears to be governed by the general law that the two substituting groups, similar or dissimilar, when in the ortho-position cause the highest, and when in the para-position the least, rotation in the dissolved ethyl tartrate. This conclusion is drawn from a comparison of the data for *o*-, *m*-, and *p*-xylene, *o*-, *m*-, and *p*-nitrotoluene, *o*- and *m*-dinitrobenzene, 2:6-dinitrotoluene, and 2:4-dinitrotoluene, whilst the behaviour of α - and β -nitronaphthalene is in accordance with it.

(3) Homogeneous ethyl tartrate has a maximum specific rotation of $+15^\circ$ at 175°C .

(4) A maximum rotation is also found in the temperature-rotation curves for solvents which raise the rotation of ethyl tartrate. In such solvents, the maximum rotation increases in value, and moves towards a lower temperature as the dilution becomes greater.

In solvents which depress the rotation of ethyl tartrate, it is probable that the maximum rotation would diminish in value and move towards a higher temperature with increasing dilution, but this has not actually been observed.

(5) The relationship between the maximum rotation and the temperature at which it occurs is much the same in different solvents, and is thus, in a sense, independent of the nature of the solvent and the concentration.

(6) The change of rotation of dissolved (or homogeneous) ethyl tartrate due to change of temperature is also nearly independent (in a proximate sense) of solvent and concentration; for example, the rotation change for a given temperature interval of a solution in acetylene tetrachloride—which has a powerful depressing effect—

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can be approximately calculated by means of a simple linear expression deduced from the behaviour of solutions in nitrobenzene—a solvent of very marked elevating influence.

EXPERIMENTAL.

Rotation of Ethyl Tartrate.

ℓ	17.8°	35.3°	60.4°	92.9°	124°	143°	160°	175.0°
α_D^{20} (100 mm.) ..	+9.222	11.174	13.318	15.022	15.774	15.926	15.88	15.74
$[\alpha]_D^{20}$	+7.64	9.39	11.45	13.28	14.35	14.75	14.95	14.99

Densities Determined.

ℓ	19.3°	33.68°	57.35°	131.2°	173.8°
d	1.2064	1.19181	1.1677	1.0919	1.0507

Ethyl Tartrate in Nitrobenzene.

I. $p = 2.00164$.

ℓ	16.1°	27.4°	39.3°	44.1°	48.1°	56.1°	78.4°	86.8°	100.0°
α_D^{20} (400 mm.) ..	+3.737	3.627	3.451	3.367	3.299	3.172	2.878	2.759	2.569
$[\alpha]_D^{20}$	+38.68	37.88	36.40	35.65	35.06	33.93	31.36	30.29	28.51

II. $p = 4.99917$.

ℓ	15.5°	20.7°	24.6°	27.7°	44.7°	50.8°	65.5°	72.5°	82.4°	87.4°	100.0°
$[\alpha]_D^{20}$ (400 mm.) ..	+8.546	8.391	8.314	8.224	7.739	7.551	7.149	6.969	6.751	6.559	6.273
$[\alpha]_D^{20}$	+35.41	34.91	34.69	34.41	32.84	32.22	30.89	30.29	29.60	28.88	27.93

III. $p = 10.0011$.

ℓ	14.2°	17.2°	19.8°	21.3°	23.3°	29.0°	43.7°	51.3°	58.7°	74.7°	82.7°	100.0°
α_D^{20} (249.6 mm.) ..	+9.509	9.499	9.484	9.459	9.409	9.289	8.932	8.757	8.534	8.134	7.939	7.365
$[\alpha]_D^{20}$	+31.53	31.58	31.60	31.55	31.43	31.18	30.35	29.96	29.55	28.39	27.9	26.27

IV. $p = 19.9446$.

ℓ	17.6°	25.3°	39.8°	44.7°	58.8°	67.3°	81.3°	100.0°
α_D^{20} (249.6 mm.) ..	+15.625	15.58	15.339	15.27	14.973	14.638	14.205	13.346
$[\alpha]_D^{20}$	+26.07	26.16	26.07	26.06	25.76	25.47	25.04	24.26

V. $p = 50.0156$.

ℓ	17.7°	32.0°	45.9°	55.1°	63.3°	71.7°	78.6°	100.0°
α_D^{20} (249.6 mm.) ..	+25.368	26.233	26.785	26.95	27.065	27.083	27.053	26.688
$[\alpha]_D^{20}$	+16.38	17.66	18.25	18.61	18.72	18.68	18.97	19.08

Densities Determined.

Solvent.		I.		II.	
t°.	d.	t°.	d.	t°.	d.
19.1°	1.20444	20.0°	1.20317	19.0°	1.20392
50.0°	1.17433	39.6°	1.18418	37.3°	1.18589
68.7°	1.1562	68.5°	1.1566	62.7°	1.1607
100.0°	1.1252	99.0°	1.1253	98.5°	1.1248

III.		IV.		V.	
t°.	d.	t°.	d.	t°.	d.
18.0°	1.20428	17.3°	1.20442	18.7°	1.20314
47.3°	1.17526	36.5°	1.18532	37.7°	1.18384
74.3°	1.1488	63.0°	1.1589	66.7°	1.1544
100.0°	1.1232	79.4°	1.1427	100.0°	1.1207
		100.0°	1.1219		

Ethyl Tartrate in o-Nitrotoluene.

 I. $p = 5.00243$.

t°	10.1°	14.7°	23.9°	39.0°	48.6°	54.1°	63.0°	67.8°	100.0°
α_D^{20} (249.6 mm.)	+5.55	5.44	5.213	4.848	4.648	4.545	4.378	4.285	3.673
$[\alpha]_D^{20}$	+37.88	37.26	35.99	33.89	32.75	32.18	31.24	30.7	27.06

 II. $p = 10.0023$.

t°	11.5°	17.5°	24.6°	38.6°	45.6°	49.4°	54.1°	61.2°	100.0°
α_D^{20} (249.6 mm.)	+9.687	9.502	9.229	8.789	8.574	8.455	8.327	8.132	6.942
$[\alpha]_D^{20}$	+32.99	32.59	31.84	30.68	30.11	29.77	29.45	28.93	25.54

 III. $p = 25.0094$.

t°	10.4°	14.6°	24.9°	38.7°	46.4°	51.9°	56.7°	66.3°	100.0°
α_D^{20} (249.6 mm.)	+18.483	18.418	18.17	17.765	17.502	17.253	17.102	16.773	15.35
$[\alpha]_D^{20}$	+25.03	25.09	24.97	24.70	24.5	24.26	24.14	23.88	22.51

 IV. $p = 50.21$.

t°	11.0°	18.5°	23.0°	40.9°	52.0°	60.4°	67.0°	100.0°
α_D^{20} (100 mm.)	+10.42	10.653	10.551	10.665	10.681	10.685	10.673	10.393
$[\alpha]_D^{20}$	+17.46	17.8	17.86	18.33	18.54	18.68	18.77	18.83

Densities Determined.

Solvent.		I.		II.		III.		IV.	
t°.	d.	t°.	d.	t°.	d.	t°.	d.	t°.	d.
16.0°	1.18742	12.0°	1.17186	14.1°	1.17129	15°	1.17509	19.4°	1.18013
40.0°	1.14472	39.5°	1.14588	42.0°	1.14456	38	1.1630	35.0°	1.16466
58.0°	1.1275	63.0°	1.1235	62.5°	1.1247	60	1.1313	57.1°	1.1427
100.5°	1.1877	101.0°	1.10871	101.0°	1.0878	101	1.0915	101.6°	1.0980

*Ethyl Tartrate in m-Nitrotoluene.*I. $p = 5.00044$.

t°	6.8°	15.0°	21.6°	46.4°	58.6°	74.6°	99.0°
α_D^{25} (249.6 mm.)	+4.232	4.178	4.15	3.878	3.763	3.62	3.347
$[\alpha]_D^{25}$	+28.96	28.78	28.74	27.40	26.88	26.2	24.74

II. $p = 9.98878$.

t°	7.8°	17.6°	28.0°	38.3°	45.1°	49.0°	69.8°	81.0°	98.8°
α_D^{25} (249.6 mm.)	+7.138	7.205	7.198	7.146	7.071	7.02	6.685	6.506	6.136
$[\alpha]_D^{25}$	+24.45	24.87	25.07	25.08	24.96	24.86	24.07	23.68	22.69

III. $p = 25.003$.

t°	7.2°	12.7°	30.3°	61.4°	79.8°	99.0°
α_D^{25} (201.5 mm.)	+10.125	10.368	10.877	11.303	11.245	11.023
$[\alpha]_D^{25}$	+17.05	17.58	18.86	19.93	20.15	20.11

IV. $p = 51.188$.

t°	6.0°	15.3°	29.1°	62.3°	81.6°	99.6°
α_D^{25} (100 mm.)	+6.938	7.575	8.25	9.213	9.465	9.563
$[\alpha]_D^{25}$	+11.38	12.52	13.80	15.86	16.58	17.02

Densities Determined.

Solvent.		I.		II.		III.		IV.	
t° .	d.	t° .	d.	t° .	d.	t° .	d.	t° .	d.
17.5°	1.1600	15.5°	1.16272	18.3°	1.16144	18.4°	1.16690	20.4°	1.1766
38.4	1.14028	40.8	1.13902	37.1	1.1437	35.6	1.15014	39.4	1.1575
56.0	1.1240	60.8	1.1203	67.2	1.1153	65.6	1.1207	63.0	1.1342
65.8	1.1147	70.7	1.1110	99.4	1.0845	99.6	1.0882	99.4	1.0976
99.6	1.0832	99.0	1.0840						

Ethyl Tartrate in p-Nitrotoluene (m. p. 54.4°; b. p. 104.5° at 9 mm. Oil-bath, 121—122°).I. $p = 20.2968$.

t°	45.5°	51.6°	65.2°	80.3°	95.0°
α_D^{25} (170 mm.)	+7.647	7.714	7.727	7.659	7.534
$[\alpha]_D^{25}$	+19.48	19.75	20.01	20.10	20.03

II. $p = 48.5$.

t°	48.9°	70.3°	95.2°
α_D^{25} (100 mm.)	+8.334	8.912	9.148
$[\alpha]_D^{25}$	+14.94	16.35	17.16

Densities Determined.

I.			II.		
t°	53.9°	72.3°	93.4°	45.7°	64.4°
d	1.1298	1.1120	1.0919	1.1487	1.1800
					77.0°
					1.1172
					97.0°
					1.0802

Ethyl Tartrate in o-Dinitrobenzene (m. p. 118°).

$p = 25.03$.

ℓ°	116.1°	134.2°	142.4°
α_D^{25} (100 mm.)	+3.52	3.216	3.098
R_D^{25} *	+35.16	32.05	30.95

* See p. 1839.

Ethyl Tartrate in m-Dinitrobenzene (m. p. 88°; b. p. 167° under 14 mm. pressure. Oil-bath, 220°).

I. $p = 24.172$.

II. $p = 49.611$.

ℓ°	81.5°	94.7°	112.0°	63.3°	89.3°	124.6°	149.2°
α_D^{25} (100 mm.)	+5.734	5.578	5.41	+9.758	9.824	9.658	9.37
$[\alpha]_D^{25}$	+18.16	17.84	17.53	+15.59	16.03	16.22	16.06

Densities Determined.

I. ℓ°	82.8°	II. 63.5°	82.5°	99.4°
d	1.3043	1.2565	1.2425	1.2251

† The pycnometer broke just after this determination, when only one observation had been made. The values for density used in calculating the specific rotation of the solution were obtained by drawing a line through the one experimental point and parallel to the density curve for the $p = 49.611$ solution. The values thus obtained are certainly not far from the truth.

Ethyl Tartrate in s-Trinitrobenzene (m. p. 123°).

I. $p = 24.933$.

ℓ°	104.0°	114.0°	129.2°	146.4°
α_D^{25} (50 mm.) ...	-1.404	-1.052	-0.594	-0.14
R_D^{25} *	-11.26	-8.44	-4.76	-1.12

* See p. 1839.

II. $p = 51.38$.

ℓ°	95.9°	117.0°	127.4°
α_D^{25} (50 mm.)	-0.40	+0.592	+1.03
R_D^{25}	-1.56	+2.30	+4.01

Owing to the high melting point of the solvent, density determinations of the solutions were not attempted. For the density of the solvent see note (p. 1842).

Ethyl Tartrate in 2:6-Dinitrotoluene (m. p. 59°).

$p = 24.753$.

ℓ°	52.1°	61.4°	77.7°	95.6°	140.2°
α_D^{25} (75 mm.)	+5.494	5.384	5.195	4.946	4.390
$[\alpha]_D^{25}$	+22.93	22.6	22.13	21.38	19.71

Densities Determined.

t°	63.3°	75.5°	80.5°
d	1.2792	1.2668	1.2616

Ethyl Tartrate in 2:4-Dinitrotoluene (m. p. 71°). $p = 24.941.$

t°	63.0°	91.3°	123.2°
α_D^{20} (75 mm.)	+3.324	3.347	3.347
$[\alpha]_D^{20}$	+13.88	14.29	14.66

Densities Determined.

t°	72.3°	82.8°	100.0°
d	1.2710	1.2604	1.2433

*Ethyl Tartrate in α -Nitronaphthalene (m. p. 58°).*I. $p = 10.214.$

t°	61.4°	73.8°	92.6°	122.8°
α_D^{20} (75 mm.)	+3.838	3.674	3.408	3.004
$[\alpha]_D^{20}$	+41.3	39.36	37.47	33.74

II. $p = 25.145.$

t°	58.8°	78.0°	96.8°	123.0°	139.0°
α_D^{20} (40 mm.)	+4.0	3.804	3.62	3.322	3.152
$[\alpha]_D^{20}$	+32.99	31.83	30.71	28.78	27.65

III. $p = 49.565.$

t°	16.7°	26.4°	47.2°	74.3°	98.8°	111.6°
α_D^{20} (40 mm.)	+6.049	6.052	5.904	5.75	5.494	5.354
$[\alpha]_D^{20}$	+24.79	24.99	24.93	24.66	24.06	23.68

Densities Determined.

I.			II.			III.		
t° 83.9°	79.0°	101.0°	62.4°	80.0°	98.7°	48.9°	61.6°	78.5°
d 1.2113	1.1986	1.1808	1.2023	1.1864	1.1698	1.2001	1.1882	1.1719

Molecular Solution-volume and Specific Rotation at 20° of Ethyl Tartrate in Various Solvents.

Solvent	p .	d 20°/4°.	M.S.V. ²⁰ , c.c.	$[\alpha]_D^{20}$.
Nitrobenzene	0	1.20356	174.0	+41.0°
	2.00164	1.20317	173.9	38.4
	4.99917	1.20293	173.0	34.9
	10.0011	1.2023	172.9	31.6
	19.9446	1.20173	172.6	26.1
	50.0156	1.20182	171.6	17.0
<i>o</i> -Nitrotoluene	0	1.16364	175.5	+42.0°
	15.00243	1.16430	175.0	36.5
	10.0023	1.16564	174.0	32.3
	25.0094	1.17029	173.0	25.0
	50.21	1.17954	172.3	17.8

Solvent.	p.	d 20°/4°.	M.S.V. ^{20°} , c.c.	[α] _D ^{20°} .
m-Nitrotoluene ...	0	1.15784	176.5	+ 34.0°
	5.00044	1.15845	175.5	28.7
	9.98874	1.15984	175.4	25.0
	25.003	1.16534	173.3	18.2
	51.188	1.1770	172.3	18.0

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CLXXXIV.—*A New Form of Gas Burette.*

By ARTHUR EDWIN HILL.

To augment the degree of accuracy to which a straight-tube gas burette can be graduated, it is necessary to enlarge the scale by increasing the length and reducing the diameter of the burette.

The advantages thereby attained are, however, more than counteracted by the disadvantages resulting from the unwieldy proportions of the apparatus, and consequent difficulty of keeping its contents at a uniform temperature.

No increased accuracy can result from a mere reduction of the diameter of the burette if unaccompanied by a proportionate decrease of its length, for its capacity is alone thereby reduced, and the percentage error on the reduced volume of gas measured retains its former value.

An attempt has been made to devise a new form of gas burette, which, although quite compact, can be graduated to a higher degree of accuracy than the ordinary form.

It is indicated in section in the figure, and consists of the vessel *AB*, made up, as shown, of a series of ten bulbs, each with a capacity of 0 c.c., connected by short, narrow tubes, on which graduations are marked. This vessel is sealed at the top to the three-way capillary tap *C*, which is especially adapted for controlling the flow of gas between the burette and the absorption pipette; this tap has already been described by the author (*Proc.*, 1908, 24, 95). The double tube *EFG* consists of the measuring tube *EF*, which is sealed to the upper end of the vessel *AB*, and has a capacity of 10 c.c. graduated into twentieths of a c.c., and the levelling tube *FG*, which serves for adjust-

ing the pressure of the gas inside the apparatus to correspond with that of the atmosphere outside.

The vessel *AB* and the side-tube *EFG* are sealed at the bottom to the three-way tap *H*, which serves to control the flow of water between the burette and the levelling reservoir connected with the lower part of the tap by the rubber tube *K*, but not shown in the figure.

The method of working is very simple, and is as follows:

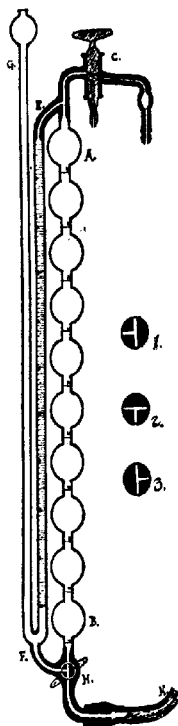
To fill the burette with water, the tap *C* is opened and the tap *H* is turned into position (1), whereby connexion is made between all parts of the burette; the reservoir is now raised until the water fills the burette and reaches the tap *C*, which is then immediately closed.

Before a sample of gas is drawn into the burette, the tap *H* is turned into position (3), whereby the double tube *EFG* is disconnected from the reservoir—at this moment it is advisable to close the opening at the top of the tube *FG* to prevent its contents from being disturbed by the partial vacuum in the vessel *AB*. When sufficient gas has entered the vessel *AB*, the tap *C* is closed, and the reservoir is raised until the water reaches the nearest graduation mark in the vessel *AB*.

The tap *H* is now slowly turned into position (2), whereby the double tube *EFG* is connected with the reservoir, the vessel *AB* disconnected from it, and a part of the gas which was compressed in the vessel *AB* is allowed to pass over into the measuring tube *EF*.

The pressure of the gas in the burette is then adjusted by lowering or raising the reservoir until the level of the water is the same in both parts of the double tube *EFG*; its volume can then be read off in twentieths of a c.c. from the combined readings of the vessel *AB* and the measuring tube *EF*. Both these readings are equally accurate, since the measuring tube *EF* has the same diameter as the short connecting tube on which the graduations of the vessel *AB* are marked.

To proceed with the analysis of the gas, the burette is connected to



an absorption pipette, the tap *H* is turned into position (1), and the gaseous contents of the burette are driven over into the pipette by opening the tap *C* and raising the reservoir. When absorption is complete, the tap *H* is turned into position (3), and the gas is returned to the vessel *AB* by lowering the reservoir, its volume being then measured in the manner already described.

The degree of accuracy to which this type of gas burette can be graduated, is limited only by the difficulty of obtaining a really sensitive pressure gauge which will give an accurate indication of the pressure of the gas in the burette. Otherwise, it would be possible to construct a burette capable of being read to hundredths of a c.c.

By applying a simple modification of the foregoing principles, it is possible to devise a very accurate volumetric burette for measuring the volume of liquids in experimental work where a high degree of accuracy is required; it is doubtful, however, whether it would be of real utility for volumetric analysis, in view of the considerable magnitude of the errors introduced by the use of indicators.

The new form of burette combines the following advantages:

- (1) It has one-tenth the number of graduations which would be required on a straight-tube gas burette with the same capacity and graduated to the same degree of accuracy.
- (2) A very open scale is provided on the measuring tube *EF*, in which one-twentieth of a c.c. is represented by a length of more than 1.5 millimetres.
- (3) The apparatus is very compact, measuring not more than sixteen inches between the taps *C* and *H*, and can therefore be fitted into a thermo-jacket of very moderate dimensions.

CONSTANTINOPLE.

CLXXXV.—*The Triazo-group. Part V. Resolution of α -Triazopropionic Acid.*

By MARTIN ONSLOW FORSTER and HANS EDUARD FIERZ.

WHILE examining the properties of α -triazocamphor, or camphoryl-azamide (Trans., 1905, 87, 826), we were impressed by the high specific rotatory power displayed by solutions in benzene, ethyl alcohol, acetone, and chloroform; with a concentration of 1 per cent., α -triazocamphor in these media gave $[\alpha]_D - 351^\circ$, -284° , -279° , and -246° respectively, values which are not approached by any known

derivative of the general formula $C_8H_{14} \begin{smallmatrix} \diagup CHX \\ \diagdown CO \end{smallmatrix}$. Moreover, whilst camphor, α -chlorocamphor, α -bromocamphor, and α -iodocamphor are dextrorotatory to the extent of $[\alpha]_D$ 42°, 96°, 135°, and 160° respectively, α -triazocamphor resembles nitrocamphor in being laevo-rotatory.

It would be reasonable to suppose from this comparison that the triazo-group is capable of exerting on the optical activity of a molecule in which it is attached to the asymmetric carbon atom an influence much more powerful than that of the halogen elements, and, in view of the general chemical resemblance between these latter and the complex in question, it became of interest to study other triazo-derivatives in which rotatory power is liable to be affected by this group. The case of α -triazocamphor being complicated by the presence of three asymmetric carbon atoms, we were led to select the simplest available molecule, namely, α -triazopropionic acid, and have resolved the racemic material (this vol., 671) by means of brucine, the salt of this alkaloid with *l*- α -triazopropionic acid crystallising readily from concentrated aqueous solutions of the racemic acid which have been neutralised with the base. Contrary to expectation, however, the influence of the triazo-group appears to be very slight, since the liquid acid has $[\alpha]_D$ -2.27°, which is raised to $[\alpha]_D$ -28.9° by dissolution in ether. This comparatively insignificant optical activity is another illustration of the wide divergence between the effect of an azoimide complex when replacing hydrogen in a cycloid system and its influence in the aliphatic series, the asymmetric triazo-compound taking a normal position among the typical α -substituted derivatives of propionic acid. Optical data concerning these are curiously incomplete and conflicting, but we believe the following to represent existing information on the subject:

Acid.	$[\alpha]_D$.	$[\alpha]_D$ in water.	$[\alpha]_D$ of Ethyl ester.
α -Chloropropionic	unknown	unknown	19.9°
α -Bromopropionic	45°	unknown	37° to 46°
α -Hydroxypropionic	unknown	2.5° to 2.0°	14.5°
α -Aminopropionic	(solid)	2.7°	less than 1°
α -Triazopropionic	2.27°	4.8° to 3.0°	18.2°

Keeping in view the tendency displayed by this class of compounds to undergo racemisation, the conditions likely to bring about this change have been, as far as possible, excluded in preparing *l*- α -triazopropionic acid, and, assuming the precautions to have been successful, it has been established that:

(a) The rotation of triazopropionic acid is very low compared with that of bromopropionic acid.

(b) The rotation of the free acid is opposite in sign to that of its ion, as in the case of lactic acid.

(c) The ester has a specific rotatory power comparable with that of lactic and chloropropionic esters, but much lower than that of bromopropionic ester.

(d) The rotation of the amide is not only much greater than that of the ester, but is opposite in sign.

(e) Replacing the triazo-complex by the amino-group leads to alanine of activity opposite to that of the original material.

The problem of referring *l*-α-triazopropionic acid to the corresponding alanine presented some difficulty at first, owing to the preclusion of alkaline reducing agents; aluminium amalgam was finally chosen, and, although no definite result was reached on reducing the ester, an aqueous solution of ammonium *l*-α-triazopropionate was found to yield *d*-alanine, a specimen of which was isolated in the form of its β-naphthalenesulphonic derivative. Through the kindness of Geheimrath Professor Emil Fischer, to whom we are indebted for several grams of *d*-alanine prepared from silk, we have been able to make a direct comparison of the β-naphthalenesulphonic derivatives from both sources, the original description (Fischer and Bergell, *Ber.*, 1902, 35, 3781) giving no information as to optical activity.

The question whether the change in direction of rotation on passing from triazopropionic acid to alanine is another case of the Walden inversion, cannot, of course, be determined until some process is devised for effecting the converse transformation.

EXPERIMENTAL.

l-α-Triazopropionic Acid, $\text{CH}_3\cdot\text{CHN}_3\cdot\text{CO}_2\text{H}$.

Preliminary experiments with cinchonine, bornylamine, and brucine indicated the last-named alkaloid as the one best calculated to effect satisfactory resolution of α-triazopropionic acid. An aqueous solution of cinchonine triazopropionate did not deposit crystals during many days, and concentration led to the separation of an oil even when conducted at the temperature of the laboratory; bornylamine formed a crystalline salt, but only in solutions so concentrated that the mother liquor was syrupy.

Two hundred and forty grams of brucine were mixed with 200 c.c. of water, and to the paste were added 70 grams of racemic α-triazopropionic acid, which quickly dissolved the alkaloid; crystallisation began almost immediately, and yielded 120 grams of brucine *l*-triazopropionate.

propionate, having $[\alpha]_D -25.6^\circ$. Recrystallisation from 120 grams of boiling water gave 90 grams of salt, which was further recrystallised from 500 c.c. of hot absolute alcohol; 70 grams of rectangular plates were thus obtained, having $[\alpha]_D -26.3^\circ$, this rotation being raised only one-tenth of a degree by another recrystallisation from 75 grams of boiling water, the final quantity of salt amounting to 62 grams. The specific rotatory power, $[\alpha]_D -26.4^\circ$, for brucine *l*-triazopropionate having been confirmed by another experiment, involving 100 grams of brucine and 30 grams of racemic α -triazopropionic acid, the specimen was regarded as an individual salt, and employed in preparing the active acid. Seventy-five grams were dissolved in warm water, treated with excess of ammonia, filtered from brucine, and extracted twice with chloroform to remove any remaining alkaloid. The aqueous solution of ammonium triazopropionate was then treated with 100 grams of sulphuric acid, diluted to 25 per cent., saturated with solid ammonium sulphate, and extracted five or six times with ether; after drying the solvent with sodium sulphate, evaporation was conducted without heating the acid, which was left in a vacuum desiccator until constant in weight, 15 grams remaining in the form of a colourless oil.

l-Triazopropionic acid has the appearance, odour, and chemical properties of the racemic substance, and the specific gravity is 1.2393 compared with water at 21° , which corresponds very nearly with that of the inactive material, 1.2247 at 25° ; in a 2-dm. tube the specimen gave $\alpha_D -5.35'$ at 21° , whence $[\alpha]_D -2.27^\circ$. A solution containing 4.1327 grams of the acid made up to 25 c.c. with ether at 23° gave $[\alpha]_D -9.33'$ in the 2-dm. tube, whence $[\alpha]_D -28.9^\circ$. The aqueous solution, however, is dextrorotatory, one containing 5.2136 grams of acid made up to 25 c.c. at 21° giving $\alpha_D 2.00'$ in the 2-dm. tube, whence $[\alpha]_D 4.8^\circ$; the same solution diluted to 50 c.c. gave $\alpha_D 0.338'$, whence $[\alpha]_D 3.0^\circ$.

The brucine salt dissolves readily in its own weight of boiling water, from which it crystallises in large, transparent, rectangular plates; it requires about five times its weight of absolute alcohol for dissolution, and is only sparingly soluble in the cold medium:

0.3183 gave 37.6 c.c. N_2 at 20° and 761 mm. $N = 13.53$.

$C_{23}H_{26}O_4N_2 \cdot C_8H_7O_2N_3$ requires $N = 13.75$ per cent.

The substance has not a definite melting point, but evolves gas at about 150° . It displays mutarotation, a solution containing 1.0005 grams made up to 25 c.c. with water giving $\alpha_D -1.50'$ in a 2-dm. tube, increasing to $-2.7'$ during half an hour on the water-bath; the initial and permanent values for the specific rotatory power are, therefore, $[\alpha]_D -22.9^\circ$ and -26.4° respectively. From this it appears that the

triazopropionic ion is dextrorotatory, and is therefore opposite in sign to the free acid, for, on calculating the contribution of the brucinium ion, this is found to be approximately $-2^{\circ}27'$, whence the contribution of the triazopropionic ion must be dextrorotatory, even supposing the undissolved salt to contain water of crystallisation, the possibility of which is suggested by the above analysis. This conclusion was confirmed by dissolving 20 grams of brucine *l*-α-triazopropionate in water, adding excess of ammonia, extracting three times with chloroform, concentrating to 100 c.c., and examining the aqueous solution in the polarimeter, when the ammonium *l*-α-triazopropionate was found to be dextrorotatory to the extent of 1° , which would correspond with an approximate value of 11° for the ion.

Ethyl l-α-Triazopropionate, $\text{CH}_3\cdot\text{CHN}_3\cdot\text{CO}_2\cdot\text{C}_2\text{H}_5$.

Slight excess of silver oxide was mixed with the active acid dissolved in ether, and, without separating the salt from unchanged oxide, considerable excess of ethyl iodide was added and left in contact, with frequent shaking, during thirty-six hours at the laboratory temperature. It was hoped in this way to avoid any risk of racemisation, and the esterification appearing to be complete, silver iodide was filtered, and a gentle current of dry air passed through the liquid while maintained under 20 mm. pressure. The limpid residue, which had the characteristic odour of the racemic ester, after remaining several days in a darkened desiccator was distilled under 5 mm. pressure, when it boiled steadily at 49° . The specific gravity was 1.063 compared with water at 22° , and the rotatory power at this temperature was $\alpha_D - 38^{\circ}42'$ in the 2-dm. tube, whence $[\alpha]_D - 18.2^{\circ}$. A 12 per cent. solution in ether had practically the same rotation, 3.0093 grams made up to 25 c.c. giving $\alpha_D - 4^{\circ}27'$ in the 2-dm. tube, whence $[\alpha]_D - 18.5^{\circ}$.

l-α-Triazopropionamide, $\text{CH}_3\cdot\text{CHN}_3\cdot\text{CO}\cdot\text{NH}_2$.

Three grams of the active ester were shaken with concentrated aqueous ammonia during six hours, becoming thereby transformed into lustrous crystals of the amide; crystallisation from benzene gave snow-white needles melting at 85° , higher by 6° than the point at which the racemic compound fuses. Although derived from the levorotatory acid, the amide is dextrorotatory, a solution containing 0.175 grams made up to 25 c.c. with water giving $\alpha_D + 49^{\circ}$ in the 2-dm. tube, whence $[\alpha]_D + 49.1^{\circ}$; 0.8986 gram made up to 20 c.c. with acetone gave $\alpha_D + 9^{\circ}$ in the same tube, whence $[\alpha]_D + 100.1^{\circ}$.

Reduction of Triazopropionic Acid to Alanine.

Many preliminary experiments with the racemic acid and its ester were directed towards replacing the triazo-group by the amino-radicle, and the selection of aluminium amalgam as reducing agent followed. Two grams of inactive α -triazopropionic ester were diluted with 50 c.c. of ether, and treated with 5 grams of amalgamated aluminium gravel added in small quantities during six hours, drops of water being introduced occasionally while the liquid was cooled externally. The ethereal solution of alanine ester was then filtered from alumina and mixed with 2 grams of picric acid in ether; the solvent, on evaporation, deposited a pale red oil, which became crystalline when stirred with a few drops of water, and, on recrystallisation from warm water, which deposited one gram of lustrous, pale yellow needles, the substance melted and decomposed at 167° :

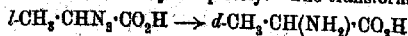
0.2310 gave 33.0 c.c. N_2 at 24° and 759 mm. $N = 16.00$.

$C_5H_{11}O_2N_3C_6H_5O_7N_3$ requires $N = 16.19$ per cent.

The melting point recorded for the picrate of ethyl α -amino-propionate by Emil Fischer is 168° .

Three grams of the *l*-ester diluted with 50 c.c. of ether were then reduced with 10 grams of aluminium amalgam, and, on concentrating the filtrate and washings to 20 c.c., the solution was found to be still levorotatory, although the rotation had diminished to about 3%. This experiment being indecisive, however, owing to the possibility of the activity being due to unattacked triazopropionic ester, an aqueous solution of ammonium *l*-triazopropionate was reduced with the same agent, which furnished an inactive solution becoming dextrorotatory on adding hydrochloric acid; the result suggested the formation of *d*-alanine, which has $[\alpha]_D 2.7^{\circ}$ in water and $[\alpha]_D 10.4^{\circ}$ in the form of hydrochloride (Fischer and Raske, *Ber.*, 1907, 40, 3720), but might equally well have arisen from unchanged *l*-triazopropionic acid, this being dextrorotatory in water. Accordingly, the liquid was neutralised with normal potassium hydroxide and agitated with excess of β -naphthalenesulphonyl chloride in ether during four hours, one molecular proportion of alkali being added after each hour; the colourless derivative obtained on acidifying the alkaline liquid with hydrochloric acid was recrystallised from boiling water, and, after being dried in the desiccator, melted at 112° . A direct polarimetric comparison between this product and the β -naphthalenesulphonic derivative of *d*-alanine was then made, the requisite amount of normal potash diluted with water being used as the solvent; in concentration approximating to 25 per cent., the specimen from *l*- α -triazopropionic acid gave $[\alpha]_D -57^{\circ}$, whilst that from *d*-alanine gave $[\alpha]_D -50.6^{\circ}$, a value perhaps a few

degrees too low because of the horny condition of the material, which it was therefore difficult to dry completely. The transformation



was thus established.

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DLXXXVI.—*The Triazo-group. Part VI. Triazoethyl Alcohol and Triazoacetaldehyde.*

By MARTIN ONSLOW FORSTER and HANS EDUARD FIERZ.

PREVIOUS communications have indicated the extent to which a carbonyl group, favourably situated in regard to the triazo-complex, is capable of modifying the behaviour of the latter towards alkalis, and it appeared to us desirable that there should be added to the positive evidence which has been already accumulated that negative testimony which an examination of triazoethyl alcohol might be expected to supply. The preparation of this material presents no difficulty, although a slight departure from the usual procedure is necessary, interaction between the halogen derivative and sodium azide being best conducted without a solvent.

Of the triazo-ketones which have been studied since the properties of α -triazocamphor first drew our attention to the subject, none has reached the standard presented by the last-named substance, namely, the quantitative character of the two-thirds nitrogen elimination by alkali. Acetonylazoimide, phenacylazoimide, 2-triazocyclohexanone, 4-triazobutanone-2, and 3-triazobutanone-2, when treated with dilute alkali, yield varying proportions of hydrazoic acid, although a largely preponderating quantity of nitrogen is lost in the elemental form, and we have suggested that the distinctive behaviour of these ketones is attributable to superior tendency towards enolisation. Triazoethyl alcohol presents a complete structural antithesis to α -triazocamphor, since the possibility of ketonisation is excluded, and we expected to find that the two-thirds nitrogen evolution would be suppressed entirely; actually, however, the course of the action is limited by the concentration of the alkali, but it is safe to assert that, under conditions which result in a vigorous decomposition of the triazo-ketones, triazoethanol remains indifferent. With solutions of alkali exceeding 40 per cent., although there is no apparent action in the cold, nitrogen and ammonia are liberated on boiling, but, as the change is accom-

panied by development of a brown coloration, it seems probable that this variation from the expected order is due to reduction by potassium triazoethoxide, and is not, therefore, a specific action of the alkali. Moreover, triazoacetaldehyde recalls the behaviour of the triazoketones, liberation of nitrogen taking place concurrently with removal of hydrazoic acid.

In an earlier paper, comment was made on the influence which the azoimide complex appears to exert in connexion with the odour of simple typical substances into which it has been introduced, for, contrary to expectation based on the properties of aromatic azoimides, the odour of those belonging to the aliphatic series proved much less intense than that of the parent substances, and was quite unmodified in the direction of anise. It is noteworthy that the odour of triazoacetaldehyde is also faint, the pungent, choking effect of acetaldehyde vapour being completely masked, whilst triazoethyl alcohol is odourless; nevertheless, the perfume of triazoethyl acetate is scarcely distinguishable from that of ethyl acetate.

During the past few years, patent literature has contained many references to the therapeutic value of substituted ethyl amino-benzoates, a notable example being diethylaminoethyl *p*-aminobenzoate, the hydrochloride of which, called novocaine by its discoverer, Einhorn, is distinguished as a non-irritant local anæsthetic. As the method for preparing novocaine is unsuited to the production of the parent base, aminoethyl *p*-aminobenzoate, or *p*-aminobenzoyloxyethylamine, we have obtained this compound by converting triazoethyl alcohol into the *p*-nitrobenzoic ester, and reducing the latter with tin and hydrochloric acid. The behaviour of the diamine is disappointing, however, and supports the recent experience of F. L. Pyma (this vol., p. 1793) in demonstrating the absence of exact knowledge concerning the relation between chemical constitution and physiological action. Dr. H. H. Dale, of the Wellcome Physiological Research Laboratories, has been so kind as to examine the hydrochloride of *p*-aminobenzoyloxyethylamine, and reports that "the 5 per cent. solution produced no perceptible loss of sensibility when applied to the eye of a rabbit: neither was there any conspicuous irritation."

EXPERIMENTAL.

Triazoethyl Alcohol (2-Triazoethanol-1), $N_3 \cdot CH_2 \cdot CH_2 \cdot OH$.

From preliminary experiments, it appeared that, although chlorine is replaced by the triazo-group when ethylene chlorohydrin is heated with aqueous sodium azide, the transformation is limited, and a more satisfactory result arises from adopting the practice of Butleroff

and Ossokin (*Annalen*, 1867, 144, 40) in the preparation of ethylene iodohydrin.

Eighty grams of dry sodium azide were heated in a Jena flask with 80 grams of ethylene chlorohydrin (0.8 mol.) on the steam-bath during forty-eight hours, the weight of liquid obtained on filtration and subsequent washing with ether being roughly that of the chlorohydrin employed; the product was heated at 100° under 70 mm. pressure during half an hour, and then distilled under a pressure of 8 mm., when 75 grams boiled steadily to the last drop at 60°:

0.1748 gave 49.5 c.c. N_2 at 23.3° and 752.5 mm., along with 0.03369 of ammonia. $N = 32.26$ as gas; 15.94 as NH_3 ; total, 48.20.

$C_2H_5ON_3$ requires $N = 48.27$ per cent.

Triazoethyl alcohol is a colourless, sweet-tasting, mobile liquid, having specific gravity 1.149 compared with water at 24°; it is miscible with water and hygroscopic, and, although without odour, the vapour when inhaled affects the blood-pressure in the manner characteristic of the triazo-group in the aliphatic series. The detonation on a hot plate is very mild. When mixed with concentrated sulphuric acid, vigorous effervescence occurs after an interval of a few seconds and stannous chloride also liberates nitrogen.

Towards aqueous potassium hydroxide the behaviour of triazoethanol depends entirely on the concentration of the alkali. At 30 per cent., the agent gives rise to very sluggish effervescence on continued boiling, the liquid remaining colourless, whilst a 70 per cent. solution, although without apparent action in the cold, develops brisk effervescence on heating, the nitrogen being accompanied by ammonia, and the liquid becoming brown. Intermediate degrees of concentration display corresponding variations between the limits indicated, from which it is clear that the attitude of triazoethanol towards alkali is quite distinct from that of the triazo-ketones, which suffer loss of nitrogen in presence of mere traces of potash. On continued heating with the above-mentioned solutions of alkali, a considerable proportion of hydrazoic acid was eliminated.

Reduction to Aminoethyl Alcohol.—The triazo-compound was treated with a 20 per cent. solution of stannous chloride in dilute hydrochloric acid until evolution of nitrogen had ceased; on adding excess of alkali, followed by benzoyl chloride, the supposed dibenzoyl derivative of aminoethyl alcohol was obtained in a viscous condition, and solidified when treated with warm water. Recrystallisation from benzene and petroleum gave lustrous, snow-white needles, melting at 88–89°:

0.2153 gave 0.5642 CO_2 and 0.1141 H_2O . $C = 71.47$; $H = 5.88$.

0.1675 „ 7.95 c.c. N_2 at 20° and 756 mm. $N = 5.40$.

$C_{15}H_{15}O_2N$ requires $C = 71.38$; $H = 5.58$; $N = 5.20$ per cent.

There is a discrepancy between the fusion-point of our specimen and that mentioned by Knorr (*Ber.*, 1897, 30, 914), who records 76°.

Triazoethyl Acetate, $\text{CH}_3\cdot\text{CO}_2\cdot\text{C}_2\text{H}_4\text{N}_3$.

Fifteen grams of triazoethyl alcohol were added to 20 grams of acetyl chloride diluted with 50 c.c. of dried chloroform, hydrogen chloride being liberated immediately; after ten minutes, the liquid was heated on the steam-bath until most of the chloroform had evaporated, and the residue, dissolved in ether, was shaken with a dilute solution of sodium carbonate. The ester was then distilled under 20 mm. pressure, when it boiled at 74°. Analysis presented the difficulties previously encountered when dealing with triazo-derivatives of the aliphatic series, and we are indebted to Mr. H. Droop Richmond for the estimation of nitrogen in the ester and its parent alcohol:

0.3490 gave 64.6 c.c. N_2 at 20° and 750.5 mm., along with 0.0444 of ammonia. $\text{N} = 21.25$ as gas; 10.49 as NH_3 ; total, 31.74.

$\text{C}_4\text{H}_7\text{O}_2\text{N}_3$ requires $\text{N} = 32.56$ per cent.

Triazoethyl acetate is a colourless, limpid liquid, with an odour much more pronounced than that of the isomeric ethyl triazoacetate, resembling more closely the perfume of ethyl acetate in both character and intensity; it has the specific gravity 1.126 compared with water at 24°. Detonation does not occur when the ester is dropped on a hot iron plate, the globules merely vaporising. Nitrogen is liberated from the substance by stannous chloride, ammonium sulphide, and concentrated sulphuric acid.

Triazoethyl p-Nitrobenzoate, $\text{NO}_2\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\cdot\text{C}_2\text{H}_4\text{N}_3$.

On mixing 9 grams of triazoethyl alcohol with 18 grams of *p*-nitrobenzoyl chloride, the temperature rose, and chloroform was therefore added, when the gently warmed solution continued to evolve hydrogen chloride during two or three hours; after spontaneous evaporation of the solvent, 15 grams of rhombic plates had separated, and recrystallisation from a moderate quantity of hot absolute alcohol yielded the ester in lustrous, yellow leaflets, melting at 82°:

0.1774 gave 36.5 c.c. N_2 at 24° and 770 mm. $\text{N} = 23.40$.

$\text{C}_9\text{H}_8\text{O}_4\text{N}_4$ requires $\text{N} = 23.73$ per cent.

The substance dissolves readily in benzene, more sparingly in alcohol or petroleum. Concentrated sulphuric acid liberates nitrogen very slowly, and stannous chloride only on heating the solution,

p-Aminobenzoylcrysthylamine, $\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CO}_2 \cdot \text{C}_2\text{H}_4 \cdot \text{NH}_2$.

Five grams of triazoethyl *p*-nitrobenzoate suspended in 50 c.c. of concentrated hydrochloric acid and 50 c.c. of absolute alcohol were treated with 15 grams of tin-foil at a temperature below 35° ; reduction was complete in thirty minutes, and, under these conditions, the amount of nitrogen liberated was negligible, although considerable effervescence takes place above 40° . It would thus appear that elimination of two-thirds the azidic nitrogen may result in producing either the element or ammonia, and a similar observation was made in using aluminium amalgam, which gave rise to torrents of ammonia and an unsatisfactory product of reduction. The liquid having been filtered through glass wool, tin was precipitated by hydrogen sulphide, the filtrate concentrated under reduced pressure to 300 c.c., and treated with lead carbonate, the filtrate from which was evaporated to half its original bulk in presence of freshly precipitated lead oxide; the last traces of hydrochloric acid were thus removed, and, on evaporating the colourless filtrate by steam and completing the concentration in a vacuum desiccator, the syrupy residue solidified to lustrous, white plates. The drained product was recrystallised from the minimum quantity of hot water, which deposited lustrous, rhombic plates, melting somewhat indefinitely at 78° ; the yield approximated to 3.5 grams:

0.2438 gave 0.4895 CO_2 and 0.1592 H_2O . $\text{C} = 54.76$; $\text{H} = 7.25$.

0.2269 „ 27.6 c.c. N_2 at 20° and 766 mm. $\text{N} = 14.06$.

$\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2 \cdot \text{H}_2\text{O}$ requires $\text{C} = 54.54$; $\text{H} = 7.07$; $\text{N} = 14.14$ per cent.

The base therefore contains water of crystallisation, and in this form is readily soluble in water and in alcohol, but insoluble in petroleum. The crystals have a faint red tinge, but they remain transparent and do not darken when exposed to air; when heated at 75° , however, the lustre is quickly lost, and by gradually raising the temperature to 105° the water of crystallisation is entirely removed;

0.1686 gave 22.9 c.c. N_2 at 21° and 768 mm. $\text{N} = 15.63$.

$\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2$ requires $\text{N} = 15.55$ per cent.

In the anhydrous condition, the substance melts at 122.5° without decomposing, shrinking slightly a few degrees below this temperature. It is readily soluble in alcohol, moderately so in acetone, and very sparingly so in boiling benzene, from which it separates in minute nodules; chloroform and petroleum appear to have no solvent action.

The hydrochloride is hygroscopic, and devoid of characteristic physiological effect.

The picrate is moderately soluble in alcohol, and crystallises in

somewhat indefinite nodules, melting at $172-175^{\circ}$, according to the rate at which the temperature is raised :

0.1631 gave 23.9 c.c. N_2 at 21° and 768 mm. $N = 16.86$.

$C_9H_{12}O_2N_2, C_8H_8O_7N_8$ requires $N = 17.11$ per cent.

$C_9H_{12}O_2N_2, 2C_8H_8O_7N_8$ " $N = 17.55$ " "

The *dibenzoyl* derivative was prepared by the Schotten-Baumann process :

0.1826 gave 12.0 c.c. N_2 at 24° and 761 mm. $N = 7.38$.

$C_{23}H_{20}O_4N_2$ requires $N = 7.22$ per cent.

The substance crystallises from acetone in silky needles, and melts at 212° without decomposing.

Triazoacetaldehyde, $N_3 \cdot CH_2 \cdot CH \cdot O$.

The chloroacetaldehyde hydrate was prepared according to the directions of Natterer (*Monatsh.*, 1882, 3, 442), and when mixed with sodium azide formed a paste which exploded mildly on heating, leaving a charred mass. Twenty grams of the hydrate were then heated at 60° with 15 grams of sodium azide in 60 c.c. of water, the clear, colourless solution becoming suddenly pink and then turbid; on the appearance of gas in small quantities, heating was discontinued, and, after forty-eight hours at the laboratory temperature, the suspended oil was extracted with ether and dried by calcium chloride. It was impossible, however, to purify the substance further. On attempting to distil under a pressure of 4 mm., an ominous rush of gas took place, containing a large proportion of hydrazoic acid, and it was therefore considered dangerous to heat the substance above 80° , at which temperature the distillation had not begun.

Although its highly unstable character precludes an extended investigation, there seems no doubt that the substance is triazoacetaldehyde. The oily liquid is heavier than water, in which it is moderately soluble, and is miscible with organic liquids; it has a faint odour recalling that of chloroacetaldehyde hydrate, and the inhaled vapour produces the throbbing sensation which has become familiar in connexion with the triazo-complex, followed by an obstinate headache suggesting that which accompanies hydrazoic acid poisoning. When preserved in the desiccator, triazoacetaldehyde quickly becomes dark yellow, and liberates hydrazoic acid, making it impossible to determine whether the oil consists of the aldehyde or its hydrate. Thrown on a hot plate, the substance explodes very mildly, in this respect resembling triazoacetone, and the behaviour of the latter is further recalled by the action of aqueous potassium hydroxide, which liberates nitrogen and ammonia in torrents, concurrently eliminating hydrazoic acid. Triazoacetaldehyde reduces Fehling's solution and

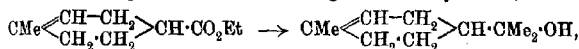
ammoniacal silver oxide immediately, but does not give an insoluble compound with sodium hydrogen sulphite; moreover, it has not been possible to isolate derivatives with hydroxylamine, phenylhydrazine, or *p*-bromophenylhydrazine, owing to the readiness with which hydrazoic acid is removed. Semicarbazide behaves in corresponding fashion, producing the characteristic bis-semicarbazone of gloxal, the change being analogous to the one by which triazoacetone is converted into the bis-semicarbazone of methylgloxal (this vol., p. 83).

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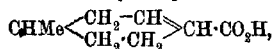
CLXXXVII.—*Experiments on the Synthesis of the Terpenes. Part I (continued). Resolution of dl-1-Methyl- Δ^1 -cyclohexene-4-carboxylic Acid and Synthesis of the Optically Active Modifications of Terpeneol.*

By KENNETH FISHER and WILLIAM HENRY PERKIN, jun.

IN Part I of this series of researches (Trans., 1904, 85, 657), it was shown that *dl*-terpineol is produced when ethyl *dl*-1-methyl- Δ^1 -cyclohexene-4-carboxylate is treated with magnesium methyl iodide,



and, since *dl*-terpineol yields *dl*-limonene (dipentane) by elimination of water and terpin by hydration, the synthesis of the whole of this important group is now complete. The next step was to attempt the synthesis of the optically active modifications of terpeneol and limonene, but, owing to the difficulty of preparing *dl*-1-methyl- Δ^1 -cyclohexene-4-carboxylic acid in quantity sufficient for resolution, the attempt had, at that time, to be abandoned. The first synthesis of an optically active menthenol and the corresponding menthadiene was subsequently realised with the aid of *dl*-1-methyl- Δ^3 -cyclohexene-4-carboxylic acid,



which was selected because it could be obtained in larger quantities than the Δ^1 -acid, and, indeed, than any other acid of this group (Trans., 1906, 89, 839).

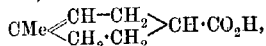
Experiment showed that this acid is readily resolved into its *d*- and *l*-modifications by the fractional crystallisation of the brucine and strychnine salts in the usual manner. The esters of the *d*- and *l*-acids

yield, on treatment with magnesium methyl iodide, the corresponding *d*- and *l*- Δ^3 -*p*-menthenols, and from the *d*-menthenol, *d*- $\Delta^{3,8}$ -*p*-menthadiene was prepared by the dehydrating action of magnesium methyl iodide. The rotatory powers of these substances are appended for comparison with the results described in the present communication.

	$[\alpha]_D$
<i>d</i> -1-Methyl- Δ^3 -cyclohexene-4-carboxylic acid	+101.1°
<i>l</i> -1-Methyl- Δ^3 -cyclohexene-4-carboxylic acid	-100.8
Ethyl <i>d</i> -1-methylcyclohexenecarboxylate	+86.5
<i>l</i> -1-methylcyclohexenecarboxylate	-83.5
<i>d</i> - Δ^3 - <i>p</i> -Menthenol(8)	+65.0 (calc.)
<i>l</i> - Δ^3 - <i>p</i> -Menthenol(8)	-67.3
<i>d</i> - $\Delta^{3,8}$ - <i>p</i> -Menthadiene	+98.2

Quite recently (Meldrum and Perkin, *Trans.*, 1908, **93**, 1417), a new method was discovered which has made it possible to prepare *dl*-1-methyl- Δ^3 -cyclohexene-4-carboxylic acid in larger quantities than before, and, in possession of about 75 grams of this acid, we have carried out a series of preliminary experiments on its resolution and conversion into the optically active terpeneols.

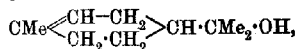
When combined with strychnine and brucine, *dl*-1-methyl- Δ^1 -cyclohexene-4-carboxylic acid,



shows the behaviour which was observed in the case of *dl*-1-methyl- Δ^3 -cyclohexene-4-carboxylic acid, that is to say, the strychnine salt, *l*BdA, is less soluble than the salt *l*BIA, whereas the solubilities of the brucine salts are in the reverse order, the salt *l*BIA being the less soluble. By taking advantage of this behaviour, we have been able to resolve *dl*-1-methyl- Δ^1 -cyclohexenecarboxylic acid into its optically active modifications, and have obtained the *d*-acid with $[\alpha]_D + 55^\circ$, and the *l*-acid with $[\alpha]_D - 58^\circ$.

Comparison with the above table shows that the rotations of the active modifications of this Δ^1 -acid are much lower than those of the corresponding Δ^3 -acid.

The esters of the *d*- and *l*- Δ^1 -acids were next prepared, and were found to have $[\alpha]_D + 50^\circ$ and -52° respectively. When these esters were treated with magnesium methyl iodide at the ordinary temperature, they were converted quantitatively into the corresponding optically active terpeneols,



which melted sharply at 35° , and had $[\alpha]_D + 44.2^\circ$ and -46.6° .

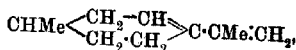
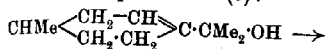
Active terpeneols of very varying rotations have been isolated from essential oils. Thus *l*-terpineol from niaouli-oil has a rotation of $[\alpha]_D - 2^\circ 10'$ (Bertrand, *Bull. Soc. chim.*, 1893, [iii], **9**, 436), whereas

Schimmel & Co. (Berichte von Schimmel & Co., 1897, 9) obtained *d*-terpineol from cardamom oil with the high rotation $[\alpha]_D + 83.31^\circ$.

Ertchikowsky (Ber., 1896, 29, 887) describes an *l*-terpineol, prepared from pinene, with a rotation as high as $[\alpha]_D - 117.5^\circ$.

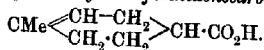
In view of the fact that the synthetical *d*- and *l*-terpineols described in the present communication have rotations of only about $[\alpha]_D \pm 46^\circ$, it is necessary that the whole matter should be systematically investigated, and experiments with this object are in progress.

We have, however, been unsuccessful in our attempts to convert these active terpeneols into the corresponding optically active limonenes. It has already been mentioned (p. 1872) that *d*- Δ^3 -*p*-menthadiene is readily obtained by the dehydrating action of magnesium methyl iodide on *d*- Δ^3 -*p*-menthenol(8):



and, as the rotation of the hydrocarbon obtained was $[\alpha]_D + 98.2^\circ$, it is unlikely that racemisation can have taken place to any considerable extent, at all events, during its formation. When the experiment was carried out under exactly the same conditions with *l*- Δ^1 -*p*-menthenol(8) (*l*-terpineol), having $[\alpha]_D - 46.6^\circ$, we were surprised to find that racemisation was almost complete, since the hydrocarbon, which was obtained in an almost quantitative yield, had a rotation of only $[\alpha]_D - 5^\circ$, and consisted essentially of dipentene. It is very remarkable that racemisation should take place to this extent during the elimination of water by the action of magnesium methyl iodide in the cold, and it is, furthermore, very difficult to find any explanation for the great difference exhibited by Δ^3 -*p*-menthenol(8) and the Δ^1 -isomeride in this respect. In order, if possible, to avoid this racemisation, we employed in the case of *d*- Δ^1 -*p*-menthenol(8) (*d*-terpineol of $[\alpha]_D + 44.2^\circ$) anhydrous oxalic acid at 100° , but racemisation was even more complete under these conditions, since the hydrocarbon obtained had a rotation of only $[\alpha]_D - 2^\circ$, and was, in fact, almost pure dipentene.

Resolution of dl-1-Methyl- Δ^1 -cyclohexenecarboxylic Acid,



In carrying out this resolution, the pure acid (25 grams) was suspended in boiling water (1.5 litres), exactly neutralised with sodium carbonate, and then mixed with a boiling concentrated alcoholic solution of the acid (25 grams) and brucine (105 grams).

The solution was then evaporated on the water-bath, with the addition from time to time of small quantities of water, until the alcohol had been removed. During this operation, the brucine salt commences to separate, and, after remaining for twenty-four hours, this was collected and decomposed by warming with excess of sodium carbonate.

The acid, which separated on acidification, had a rotation of $[\alpha]_D - 24.7^\circ$, and was treated again with sodium carbonate and brucine under exactly the same conditions. After repeating the operation several times, an acid was obtained, which, on crystallisation from light petroleum, melted at about 99° , and had the rotation $[\alpha]_D - 58^\circ$.

Since the experiment preceding this had yielded an acid of rotation $[\alpha]_D - 55^\circ$, it did not seem probable that further treatment with brucine and sodium carbonate would alter the rotation to any considerable extent. The acids of low rotation obtained during the intermediate stages of the resolution were many times systematically treated with sodium carbonate and brucine in order to extract as much of the *l*-acid as possible. In this way, a considerable quantity of the *l*-acid accumulated, which had a rotation of about $[\alpha]_D + 28^\circ$, and this was now subjected to repeated treatment with sodium carbonate and strychnine. Since strychnine and the strychnine salt of the acid are much less soluble in dilute alcohol than brucine or the brucine salt, it was necessary to employ more alcohol, and to be careful that the strychnine used was freshly precipitated and in as fine a state of division as possible. By repeating the resolution with strychnine a large number of times, an acid was ultimately obtained which melted at about 99° , and had $[\alpha]_D + 55^\circ$:

0.1388 gave 0.3485 CO_2 and 0.1077 H_2O . $\text{C} = 68.5$; $\text{H} = 8.7$.

$\text{C}_8\text{H}_{12}\text{O}_2$ requires $\text{C} = 68.6$; $\text{H} = 8.6$ per cent.

Synthesis of d- and l-Terpineol, $\text{CMe} \begin{smallmatrix} \text{CH}-\text{CH}_2 \\ \text{CH}_2-\text{CH}_2 \end{smallmatrix} \text{CH} \cdot \text{CMe}_2 \cdot \text{OH}$.

In carrying out the synthesis of *l*-terpineol, ethyl *l*-1-methyl- Δ^1 -cyclohexenecarboxylate was first prepared by leaving the acid (20 grams of rotation -58°) in contact with alcohol (150 c.c.) and sulphuric acid (10 c.c.) at the ordinary temperature for four days. The product was dissolved in two volumes of ether, mixed with water, the ethereal solution well washed with dilute sodium carbonate, dried, and fractionated, when the whole quantity passed over at $145-147^\circ/100$ mm., the yield being almost quantitative:

0.1483 gave 0.3880 CO_2 and 0.1296 H_2O . $\text{C} = 71.3$; $\text{H} = 9.7$.

$\text{C}_{10}\text{H}_{18}\text{O}_2$ requires $\text{C} = 71.4$; $\text{H} = 9.5$ per cent.

Obtained in this way, ethyl *l*-1-methyl- Δ^1 -cyclohexenecarboxylate had a

rotation of $[\alpha]_D - 52^\circ$. In order to prepare the corresponding *l*-terpineol, the ester (20 grams) was gradually added to an ethereal solution of magnesium methyl iodide (containing 11 grams of magnesium), care being taken to avoid any rise of temperature.

After twenty-four hours, the product was decomposed by water and dilute hydrochloric acid, the ethereal solution washed well, evaporated, and the residual oil mixed with alcoholic potash (KOH = 5 grams) and left for twenty-four hours. Water was then added, the oil extracted with ether, the ethereal solution washed, dried, and evaporated, and the residue fractionated, when almost the whole quantity passed over at $109-110^\circ$ (15 mm.) and, on remaining in the ice-chest for a few days, became almost solid. The mass was left in contact with porous porcelain until quite dry, and then analysed:

0.1016 gave 0.2883 CO_2 and 0.1050 H_2O . $\text{C} = 77.4$; $\text{H} = 11.5$.

$\text{C}_{10}\text{H}_{18}\text{O}$ requires $\text{C} = 77.9$; $\text{H} = 11.7$ per cent.

This synthetical *l*-terpineol possessed the characteristic odour of lilac, melted sharply at 35° , and had $[\alpha]_D - 46.6^\circ$.

After a preliminary experiment had shown that *dl*-terpineol is converted almost quantitatively into dipentene when it is left in contact with an excess of magnesium methyl iodide for four days, this method of elimination of water was tried in the present case, as it was thought that, by working at the ordinary temperature, risk of racemisation would be avoided. *l*-Terpineol (20 grams) was gradually added to an ethereal solution of magnesium methyl iodide containing 12 grams of magnesium, and, after remaining for four days at the ordinary temperature, the product was decomposed by water and dilute hydrochloric acid, the ethereal solution washed well, dried, evaporated, and the oil fractionated, when almost the whole quantity passed over at $179-180^\circ/760$ mm. After distillation over sodium, this hydrocarbon had a rotation of only $[\alpha]_D - 5.2^\circ$, and examination showed that it consisted essentially of dipentene.

In the experiments on the synthesis of *d*-terpineol, *d*-1-methyl- ϵ -cyclohexenecarboxylate was prepared from the *d*-acid ($[\alpha]_D + 55^\circ$) in the manner described in the case of the *l*-modification, and found to distil at $145-147^\circ/100$ mm:

0.1915 gave 0.4975 CO_2 and 0.1660 H_2O . $\text{C} = 70.9$; $\text{H} = 9.6$.

$\text{C}_{10}\text{H}_{16}\text{O}_2$ requires $\text{C} = 71.4$; $\text{H} = 9.5$ per cent.

The specimen of ethyl *d*-1-methyl- Δ^1 -cyclohexenecarboxylate obtained in this way had a rotation of $[\alpha]_D + 50^\circ$. In preparing *d*-terpineol, this ester (15 grams) was treated with magnesium methyl iodide, containing 10 grams of magnesium, in the manner described in the case of the *l*-modification. It distilled at $109-110^\circ/15$ mm., and, when left in the ice-chest, gradually crystallised. The crystals, after

draining on porous porcelain, melted at 33—35°, and had $[\alpha]_D +44.2^\circ$. In the hope of obtaining *d*-limonene by the elimination of water, *d*-terpineol (10 grams) was heated with anhydrous oxalic acid (15 grams) on the water-bath for six hours. The yellow product was mixed with water, extracted with ether, the ethereal solution dried, evaporated, and the residue fractionated, when about one-third distilled at 112—114°/100 mm. This hydrocarbon had a rotation of only $[\alpha]_D -2^\circ$, so that racemisation had not only been complete, but the sign had apparently also been changed to a slight extent. Examination showed that this hydrocarbon was almost pure *d*-pentene. The high boiling residue in the distilling flask consisted, in part at least, of the oxalic ester of terpineol, and this residue was found to be also inactive.

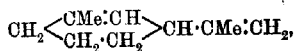
The authors wish to thank Messrs. F. W. Kay and W. N. Haworth for valuable assistance, and they also wish to state that much of the heavy expense of this investigation was covered by grants from the Research Funds of the Royal Society and the Chemical Society.

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CLXXXVIII.—*Experiments on the Synthesis of the Terpenes. Part XIII. Synthesis of isoCarvestrene ($\Delta^{6,8(9)}$ -m-Menthadiene) and its Derivatives.*

By KENNETH FISHER and WILLIAM HENRY PERKIN, jun.

DURING last year (Perkin and Tattersall, *Trans.*, 1907, 91, 480) an account was published of a series of experiments which resulted in the synthesis of carvestrene,

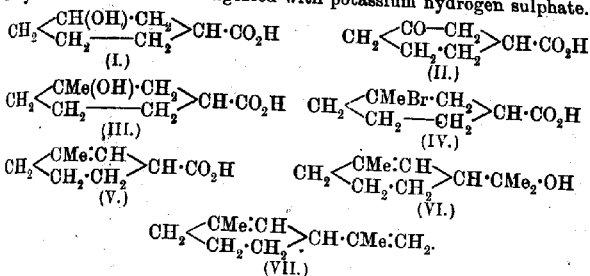


and several of its more important derivatives, and, as the methods employed during that research have a direct bearing on the present communication, the course of that synthesis may be briefly sketched in the following way. In the first place, *m*-hydroxybenzoic acid was reduced by sodium and alcohol to *cyclohexanol-3-carboxylic acid* (I), which, on oxidation with chromic acid, yielded *cyclohexanone-3-carboxylic acid* (II). By the action of magnesium methyl iodide on the ester of this acid, 1-methylcyclohexan-1-ol-3-carboxylic acid (III)

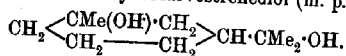
was obtained, and was next converted by treatment with hydrobromic acid into 1-bromo-1-methylcyclohexane-3-carboxylic acid (IV).

When this bromo-acid was digested with pyridine, it was decomposed with elimination of hydrogen bromide and formation of 1-methyl- Δ^1 -cyclohexene-3-carboxylic acid (V), the ester of which was converted into Δ^1 -*m*-menthenol(8) or dihydrocarvestrenol (VI) by the action of magnesium methyl iodide.

$\Delta^{1,8(9)}$ -*m*-Menthadiene or carvestrene (VII) was finally obtained when dihydrocarvestrenol was digested with potassium hydrogen sulphate.



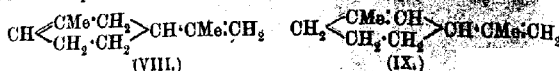
The properties of carvestrene which have a direct bearing on the present communication are the following. Carvestrene distils at 79°, gives a violet coloration when sulphuric acid is added to its solution in acetic anhydride, and combines with hydrogen chloride and hydrogen bromide to form carvestrene *trans*-dihydrochloride, $\text{C}_{10}\text{H}_{16}\cdot 2\text{HCl}$ (m. p. 52·5°), and *trans*-dihydrobromide, $\text{C}_{10}\text{H}_{16}\cdot 2\text{HBr}$ (m. p. 48—50°), respectively. Dihydrocarvestrenol, $\text{C}_{10}\text{H}_{17}\cdot\text{OH}$, distils at 105—108°/30 mm., and is converted by shaking with dilute sulphuric acid into *cis*-tetrahydrocarvestrenediol (m. p. 94°),



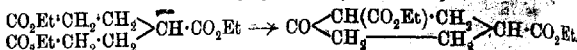
The corresponding *trans*-modification, obtained from *trans*-carvestrene dihydrobromide by treatment with silver acetate and subsequent hydrolysis, melts at 127°. A direct comparison of these synthetical products with the actual specimens of carvestrene and its derivatives prepared by Baeyer (*Ber.*, 1894, 27, 3486) during his classical researches on this hydrocarbon, proved conclusively that they are identical.

With the single exception of sylvestrene, the constitution of which still remains to be proved, the terpenes of the *m*-menthadiene series do not appear to occur in essential oils, and, as they possess many properties of great interest, it seemed important to attempt to prepare some, at all events, of the unknown members, by synthetical means. The present communication contains an account of the synthesis of

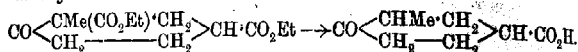
$\Delta^4,8,9$ -*m*-menthadiene (VIII), for which, on account of its very close relationship to carvestrene (IX), we propose the name *isocaradiene*:



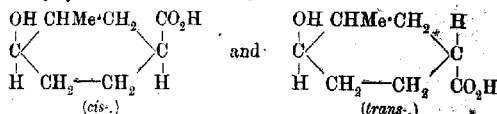
A short time since (Kay and Perkin, Trans., 1906, 88, 1647) it was shown that ethyl cyclohexanone-2:4-dicarboxylate is readily obtained when sodium reacts with ethyl pentane- α,γ,ϵ -tricarboxylate:



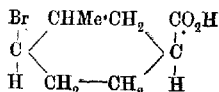
We now find that the sodium derivative of this ester is converted quantitatively by treatment with methyl iodide into *ethyl 1-methylcyclohexan-6-one-1:3-dicarboxylate*, and that this ester is decomposed on hydrolysis with formation of *1-methylcyclohexan-6-one-3-carboxylic acid*:



This new keto-acid melts at 94–96°, yields an *oxime* (m. p. 172°) and a *semicarbazone* (m. p. 200°), and, when reduced by sodium amalgam, is converted into a mixture of the *cis*- and *trans*-modifications of *1-methylcyclohexan-6-ol-3-carboxylic acid*:



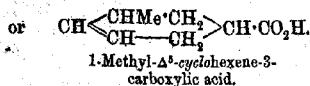
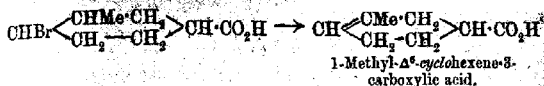
in which the latter greatly predominates. The *cis*-acid melts at 141°, yields a *lactone* (m. p. 46°), and, when treated with hydrobromic acid, is converted into *cis-6-bromo-1-methylcyclohexane-3-carboxylic acid* (m. p. 53°):



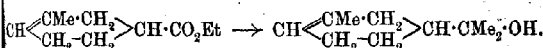
The *trans*-hydroxy-acid melts at 115–117°, and also reacts readily with hydrobromic acid, but the *trans-6-bromo-1-methylcyclohexane-3-carboxylic acid* thus produced is a syrup.

When the mixture of the *cis*- and *trans*-modifications of *6-bromo-1-methylcyclohexane-3-carboxylic acid*, obtained by the action of hydrobromic acid on the product of the reduction of *1-methylcyclohexan-6-one-3-carboxylic acid*, is esterified and digested with diethylaniline, hydrogen bromide is eliminated with the formation of *ethyl 1-methyl- Δ^6 -cyclohexene-3-carboxylate* (b. p. 146°/100 mm.), which on hydrolysis yields the free acid (b. p. 184°/100 mm.). The elimination of hydrogen

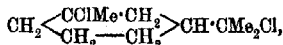
bromide from 6-bromo-1-methylcyclohexanecarboxylic acid may take place in the two following directions:



In order to decide between these two possibilities, a long series of oxidation experiments with ozone, permanganate, and nitric acid was undertaken, but the products in all these cases were syrups which we were unable to purify. We found, however, that the syrupy product obtained when the unsaturated acid was treated with ozone is a tetrionic acid, and this is strong evidence in favour of the presence of the group $-\text{CH}:\text{CMe}-$. Fortunately, as the research developed, we were able to obtain indirect evidence which conclusively demonstrated that the product of the elimination of hydrogen bromide from 6-bromo-methylcyclohexane-3-carboxylic acid is 1-methyl- Δ^4 -cyclohexene-3-carboxylic acid. Furthermore, the fractional crystallisation and analysis of the calcium salt of the unsaturated acid (p. 1887) proved that it did not contain the other isomeride, even in small quantities. When ethyl 1-methyl- Δ^4 -cyclohexene-3-carboxylate is added to an aqueous solution of magnesium methyl iodide, it is quantitatively converted into *dihydroisocarvestrenol* or Δ^6 -*m-menthenol*(8):

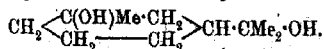


This interesting substance has the penetrating odour of peppermint and terpineol characteristic of its class; it distils at $108^\circ/20$ mm., and yields a *nitrosochloride*, melting at 125° . It reacts also with hydrogen chloride and bromide, yielding a *dihydrochloride*, $\text{C}_{10}\text{H}_{16} \cdot 2\text{HCl}$, and a *dihydrobromide*, $\text{C}_{10}\text{H}_{16} \cdot 2\text{HBr}$, which melt at 52.5° and $48-49^\circ$ respectively, and careful comparison has shown that these halogen derivatives are identical with carvestrene dihydrochloride,



and carvestrene dihydrobromide.

Furthermore, dihydroisocarvestrenol is converted by the prolonged action of dilute sulphuric acid into *cis-tetrahydrocarvestrendiol* (m. p. 94°)



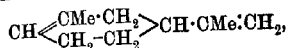
These important reactions prove in a striking manner the position

of the double linking, not only in dihydroisocarvestrenol, but also in 1-methyl- Δ^6 -cyclohexene-3-carboxylic acid.

Remarkable results were obtained during the investigation of the products of the elimination of water from dihydroisocarvestrenol.

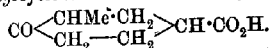
This elimination takes place very readily when the tertiary alcohol is left in contact with a large excess of magnesium methyl iodide at the ordinary temperature, and results in the formation of isocarvestrene (b. p. 176—177°/765 mm.) and a diterpene, $C_{30}H_{48}$, b. p. 188—190°/20 mm., which we have named diisocarvestrene.

isocarvestrene has been very carefully investigated, and it is pointed out in the experimental part (p. 1891) that it exhibits quite unexpected properties both from a physical and chemical point of view. The determination of its refractive power has yielded results which are very high, and agree better with those characteristic of a terpene containing conjugated double linkings than with those of the hydrocarbon of the formula



which should result from the elimination of water from dihydroisocarvestrenol. Another remarkable point is the fact that, although dihydroisocarvestrenol reacts so readily with halogen acids to yield derivatives of carvestrene, these derivatives are not produced by the action of halogen acids on isocarvestrene. The hydrocarbon on the contrary, appears to behave towards bromine and halogen acid in the way characteristic of a terpene containing conjugated double linkings (compare p. 1891).

1-Methylcyclohexan-6-one-3-carboxylic Acid,



The starting point in the synthesis of this keto-acid was ethyl cyclohexanone-2:4-dicarboxylate, which was prepared by the action of sodium on ethyl pentane- α - γ -tricarboxylate by the method described by Kay and Perkin (Trans., 1906, 89, 1647). This keto-ester (24 grams) was mixed with a solution of sodium (2.3 grams) in methyl alcohol, cooled well, and then methyl iodide (17 grams) gradually added. After remaining overnight, the product, which was quite neutral, was mixed with water, extracted with ether, the ethereal solution washed with water, evaporated, and the residual oil hydrolysed by boiling with dilute sulphuric acid (10 per cent.) for several hours and until the evolution of carbon dioxide had completely ceased. The product was saturated with ammonium sulphate, repeatedly extracted with ether, the ethereal solution dried and evaporated, when a 37

remained which, especially when rubbed with a rod, rapidly crystallised. The mass was left in contact with porous porcelain until free from oil, and the colourless residue recrystallised from ether. A further quantity of solid was obtained by extracting the porous porcelain in a Soxhlet apparatus and fractionating the extract, when a quantity of oil distilled at 190–200°/20 mm. and crystallised on cooling:

0.1520 gave 0.3438 CO_2 and 0.1060 H_2O . $\text{C} = 61.7$; $\text{H} = 7.7$.

$\text{C}_8\text{H}_{12}\text{O}_2$ requires $\text{C} = 61.5$; $\text{H} = 7.7$ per cent.

1-Methylcyclohexan-6-one-3-carboxylic acid melts at 93–94°, and is sparingly soluble in dry ether or light petroleum; it dissolves easily in alcohol, benzene, chloroform, or hot water, but is much less soluble in cold water. It crystallises from ether in crusts, from a mixture of benzene and light petroleum in groups of irregular plates, and from water in hard, glistening, six-sided plates.

In order to determine whether the acid (like the parent substance, cyclohexanone-4-carboxylic acid: *Trans.*, 1904, 85, 426) crystallises with water, a specimen was crystallised from water, and, after remaining in the air for several days, the analysis:

0.1453 gave 0.3279 CO_2 and 0.1038 H_2O . $\text{C} = 61.5$; $\text{H} = 7.9$ per cent.

showed that the acid was anhydrous. The basicity of the acid was determined by titration with decinormal sodium hydroxide, when 0.0775 neutralised 5.0 c.c., whereas this amount of a monobasic acid, H_{12}O_2 , should neutralise 4.97 c.c. The neutral solution of the ammonium salt of the acid showed the following behaviour with reagents. The addition of barium or calcium chlorides, or lead or copper acetates, gave no precipitate even on boiling, but, on the addition of silver nitrate, the silver salt separated as a crystalline precipitate, which is readily soluble in hot water, and separates, on cooling, as a satiny mass of crystals somewhat like silver acetate.

The oxime.—This derivative was prepared by dissolving the acid in excess of aqueous potassium hydroxide and adding hydroxylamine hydrochloride.

After two days, the solution was acidified with dilute hydrochloric acid, when a white, chalky precipitate separated, which was collected and crystallised from water:

0.1372 gave 9.5 c.c. N_2 at 16° and 762 mm. $\text{N} = 8.1$.

$\text{C}_8\text{H}_{12}\text{O}_2\text{N}$ requires $\text{N} = 8.2$ per cent.

This oxime softens at about 165°, and melts at 171–172°; it is rather sparingly soluble in cold, but readily so in hot, water, and separates, when the solution is allowed to cool slowly, in glistening needles grouped in stars.

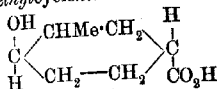
The semicarbazone separates at once as a granular precipitate when

the dilute aqueous solution of the acid is mixed with sodiumcarbazide hydrochloride and sodium acetate. The precipitate was collected, washed, and recrystallised from much water, from which it is deposited in minute crystals; it may also be crystallised from alcohol, in which it is sparingly soluble. When rapidly heated, it softens at 195° and melts at about 200° with decomposition:

0.1626 gave 27.8 c.c. N_2 at 16° and 762 mm. $N = 20.0$.

$C_9H_{15}O_3N_3$ requires $N = 19.7$ per cent.

trans-1-Methylcyclohexan-6-ol-3-carboxylic Acid,



In preparing this acid, 1-methylcyclohexan-6-one-3-carboxylic acid is dissolved in as little sodium carbonate as possible, and treated, in a wide bottle fitted with a mechanical stirrer, with three times the theoretical amount of freshly-prepared sodium amalgam (3 per cent.).

The amalgam was added in about six portions during eight hours, and hydrochloric acid run in from time to time in order to neutralise most of the alkali as it was formed, but the solution was always kept distinctly alkaline. The product was acidified, extracted ten times with ether on the machine, the ethereal solution dried, evaporated, and the residual syrup again reduced in the same way.* After the second reduction and extraction, the syrupy acid generally became semi-solid, and, in contact with porous porcelain, a colourless mass was obtained, which, after crystallisation from ether, gave the following results on analysis:

0.1377 gave 0.3067 CO_2 and 0.1115 H_2O . $C = 60.7$; $H = 9.0$.

$C_8H_{14}O_3$ requires $C = 60.8$; $H = 8.8$ per cent.

This formula was controlled by titration with decinormal sodium hydroxide, when 0.0796 required 5.05 c.c. for neutralisation, whereas this amount of a monobasic acid, $C_8H_{14}O_3$, should neutralise 5.04 c.c.

trans-1-Methylcyclohexan-6-ol-3-carboxylic acid melts at $115-117^{\circ}$ is sparingly soluble in cold, but much more readily so in boiling ether, and separates when the solution is concentrated and allowed to stand, in hard crusts. It appears to be more soluble in warm water than the *cis*-acid, and, unlike the latter, it usually takes several hours to crystallise, and then separates in stellate group of flat needles. When the *trans*-hydroxy-acid (0.2 gram) is heated in a test-tube, it melts, and then distils without the least indication of

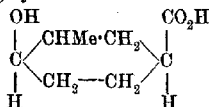
* If the amalgam employed is prepared from freshly-distilled mercury and clean sodium, this second reduction is often unnecessary.

elimination of water or charring. In contact with a speck of the crystalline hydroxy-acid, the colourless, syrupy distillate rapidly solidifies, melts directly at $108-113^{\circ}$, and consists of the almost pure *trans*-acid. This experiment shows that the *trans*-acid distills under the ordinary pressure without the formation apparently even of traces of the lactone of the *cis*-acid.

trans-6-Bromo-1-methylcyclohexane-3-carboxylic Acid.—The *trans*-hydroxy-acid dissolves readily in fuming aqueous hydrobromic acid (saturated at 0°), and the solution remains clear, but, when gradually heated on the water-bath, it clouds and an oil separates. Since this oil did not crystallise even on long standing, water was added, the bromo-acid extracted with ether, the ethereal solution dried and evaporated, when a syrup was obtained which contained about 34 per cent. of bromine, whereas the formula $C_8H_{15}O_2Br$ requires 36.2 per cent.

In this behaviour with hydrobromic acid, the *trans*-acid differs, in a striking manner, from the corresponding *cis*-acid (p. 1885).

cis-1-Methylcyclohexan-6-ol-3-carboxylic Acid,



and its Lactone.

It has already been mentioned (p. 1878) that the product of the reduction of 1-methylcyclohexan-6-one-3-carboxylic acid contains both the *cis*- and *trans*-modifications of 1-methylcyclohexanolcarboxylic acid, but that the *trans*-modification greatly predominates. The separation of the *cis*-acid proved, therefore, to be a matter of difficulty, and it was ultimately accomplished by the following indirect process.

The crude mixture of hydroxy-acids was dissolved in 5 volumes of hydrobromic acid (saturated at 0°), and then heated on the water-bath until the separation of the oily bromo-acids was complete. Water was then added, the bromo-acids extracted with ether, the ethereal solution washed, dried, evaporated, and the residue heated to boiling with 3 volumes of anhydrous pyridine for two hours. The product was acidified and distilled in steam, when an oil (*A*) passed over, and, as soon as this ceased to condense, the flask was changed and the distillation continued so long as a distinctly acid distillate (*B*) passed over. The residue in the distillation flask yielded, on extraction with ether, considerable quantities of the *trans*-hydroxy-acid. The distillate (*A*) was extracted with ether, the ethereal solution dried and evaporated, and the oily residue left in the ice-chest, when,

After some days, a quantity of crystals of the lactone of the *cis*-acid had separated and were collected. The filtrate, which consisted largely of methyl- Δ^6 -cyclohexene-3-carboxylic acid, was converted into the ester of the latter, and employed as such in the preparation of dihydroscarcostrenol (p. 1887). The lactone of the *cis*-acid was left in contact with porous porcelain until quite free from oil, and then crystallised from light petroleum (b. p. 35–40°):

0.1745 gave 0.4393 CO_2 and 0.1342 H_2O . $\text{C} = 68.8$; $\text{H} = 8.5$.
 $\text{C}_8\text{H}_{12}\text{O}_2$ requires $\text{C} = 68.6$; $\text{H} = 8.6$ per cent.

The lactone of *cis*-1-methylcyclohexan-6-ol-3-carboxylic acid melts at 46–47°, and is readily soluble in most organic solvents, but sparingly so in cold light petroleum. When boiled with water, it is hydrolysed only with great difficulty, and it is also only slowly attacked by dilute sodium carbonate. It dissolves readily in hot dilute potassium hydroxide, and, on acidifying and extracting with ether, a solid acid is obtained which, after recrystallisation from water, melts at 139–141° and consists of pure *cis*-1-methylcyclohexanolcarboxylic acid. The lactone dissolves in fuming hydrobromic acid (saturated at 0°), but decomposition takes place immediately; the solution clouds and an oil separates on the surface of the acid, which sometimes crystallises at once and always in a few hours, and the mass, after contact with porous porcelain, melts at about 53° and consists of *cis*-6-bromo-1-methylcyclohexane-3-carboxylic acid (compare p. 1885).

For the purpose of examination, some of the *cis*-methylcyclohexanolcarboxylic acid was obtained, as just explained, by the hydrolysis of the lactone, but the larger quantity was prepared from the steam distillate (B) (p. 1883). This was neutralised with sodium carbonate, evaporated to a small bulk, acidified, saturated with ammonium sulphate, and repeatedly extracted with much ether on the machine.

After drying thoroughly and concentrating considerably, the crystalline acid commenced to deposit from the boiling solution, and, on standing, hard crusts separated:

0.1705 gave 0.3789 CO_2 and 0.1342 H_2O . $\text{C} = 60.6$; $\text{H} = 8.7$.
 $\text{C}_8\text{H}_{14}\text{O}_3$ requires $\text{C} = 60.8$; $\text{H} = 8.8$ per cent.

The basicity of the acid was determined by titration with decinormal sodium hydroxide, when 0.0930 required 5.87 c.c. for neutralisation, whereas this amount of a monobasic acid, $\text{C}_8\text{H}_{14}\text{O}_3$, should neutralise 5.89 c.c.

cis-1-Methylcyclohexan-6-ol-3-carboxylic acid melts at about 140–141°. It is comparatively sparingly soluble in cold water, but dissolves readily on warming, and separates, when the solution is allowed to cool slowly, in compact, stellate groups of needles.

When the *cis*-acid is gently heated in a test-tube, it effervesces

owing to the escape of steam and an oil distils which, on cooling, crystallises and consists of the lactone, since, after contact with porous porcelain, it melts at about 40° and is insoluble in cold sodium carbonate.

A curious result was obtained in an attempt to prepare the ester of the *cis*-acid. The acid (6 grams) was digested with alcohol (32 grams) and sulphuric acid (8 grams) for several hours on the water-bath, the product extracted in the usual way and fractionated, when an oil distilled at about $152^{\circ}/100$ mm. and gave the following results on analysis:

0.1110 gave 0.2854 CO_2 and 0.0959 H_2O . $\text{C} = 70.1$; $\text{H} = 9.6$.

The ester of the *cis*-acid requires $\text{C} = 64.5$; $\text{H} = 9.7$ per cent., whereas the ester of 1-methyl- Δ^6 -cyclohexene-3-carboxylic acid (compare p. 1887) contains $\text{C} = 71.4$; $\text{H} = 9.6$ per cent., and distils at about $146^{\circ}/100$ mm. There can therefore be no doubt that the oil obtained by the esterification of the *cis*-acid, under the above conditions, consists essentially of ethyl methylcyclohexenecarboxylate, water having been eliminated during the preparation. For the sake of comparison, the *trans*-hydroxy-acid was esterified under exactly the same conditions, and found to yield an ester distilling at about $155\text{--}159^{\circ}/30$ mm.

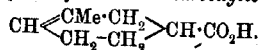
cis-6-Bromo-1-methylcyclohexane-3-carboxylic Acid.—This acid is most readily obtained from the lactone by the action of hydrobromic acid (p. 1884), but a considerable quantity was prepared directly from the *cis*-hydroxy-acid. This acid dissolves readily in fuming hydrobromic acid (saturated at 0°); the solution clouds almost immediately, and a syrup separates which sometimes crystallises at once, but usually only after several hours. The crystals were collected, and left in contact with porous porcelain over solid potassium hydroxide until quite dry:

0.1265 gave 0.1054 AgBr . $\text{Br} = 35.5$.

$\text{C}_8\text{H}_{13}\text{O}_2\text{Br}$ requires $\text{Br} = 36.2$ per cent.

cis-6-Bromo-1-methylcyclohexane-3-carboxylic acid melts at about 53° , and is readily soluble in most solvents; it may be crystallised from formic acid, but with considerable loss.

1-Methyl- Δ^6 -cyclohexene-3-carboxylic Acid,



This acid is produced, together with the lactone of *cis*-1-methylcyclohexan-6-ol-3-carboxylic acid, when crude 6-bromo-1-methylcyclohexane-3-carboxylic acid is digested with pyridine (p. 1884), but it is most conveniently prepared by the following process.

in contact with porous porcelain in the air for several days and then analysed:

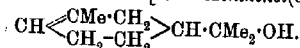
0.4108, heated at 130° until constant, lost 0.0850 H₂O and gave 0.1403 CaSO₄. H₂O = 20.7; Ca = 10.3.

(C₁₀H₁₆O₂)₂Ca, 4½ H₂O requires H₂O = 20.3; Ca = 10.0 per cent.

The mother liquor from this crystallisation was concentrated, and two other crops of calcium salt collected; these were found to have exactly the same appearance as the first crop, and to contain 21.0 and 20.9 per cent. of water of crystallisation respectively.

The filtrate from the last crop was acidified, and the acid (9 grams), which distilled at 184–186°/100 mm., again converted into calcium salt, when exactly similar crystals were obtained, which contained 20.9 per cent. of water. We therefore conclude that the unsaturated acid, prepared by the above process, consists entirely of 1-methyl-Δ⁶-cyclohexene-3-carboxylic acid, and does not contain even traces of another isomeride.

Dihydroisocarvestrenol [Δ⁶-m-Menthenol(8)],



The ethyl 1-methyl-Δ⁶-cyclohexene-3-carboxylate required for this synthesis was prepared by dissolving the pure acid (20 grams) in 10 per cent. alcoholic sulphuric acid (150 c.c.) and allowing the solution to remain at the ordinary temperature for five days.

It was isolated in the usual manner, and distilled at 146°/100 mm.

This ester (27 grams) was added to an ethereal solution of magnesium methyl iodide (containing 12 grams Mg), when little evolution of heat was noticed at first, but, on standing, the temperature of the ether gradually rose to the boiling point. After four hours, the product was cautiously decomposed by water and dilute hydrochloric acid, the ethereal solution separated, washed with very dilute sulphurous acid to remove iodine, evaporated, and the residue mixed with potassium hydroxide (4 grams), dissolved in methyl alcohol, and allowed to stand for twelve hours in order to remove any unchanged ester which might be present.

Water was then added, the oil extracted with ether, the ethereal solution well washed, dried and evaporated, and the residue fractionated under reduced pressure:

0.1648 gave 0.4710 CO₂ and 0.1755 H₂O. C = 77.7; H = 11.8.

0.1452 „ 0.4125 CO₂ „ 0.1551 H₂O. C = 77.5; H = 11.9.

C₁₀H₁₈O requires C = 77.9; H = 11.7 per cent.

Dihydroisocarvestrenol boils at 106–107°/20 mm., and is a rather viscous oil with a pungent odour of terpineol. When a drop of

sulphuric acid is added to its solution in acetic anhydride, a violet coloration is produced, which soon becomes an intense methylene-blue and, on longer standing, again violet.

Density: $d_{20^{\circ}/20^{\circ}} = 0.9376$; $d_{20^{\circ}/4^{\circ}} = 0.9359$.

Refractive power at 20° :

	μ	$\frac{\mu-1}{d}$	$\frac{\mu-1}{d} \cdot p$	$\frac{\mu^2-1}{\mu^2+2} \cdot \frac{p}{d}$
α	1.47751	0.51022	78.573	46.54
β	1.48711	0.52047	80.152	47.83
γ	1.49312	0.52688	81.189	47.83

Dispersion $\gamma - \alpha = 2.566$.

These physical constants agree closely with those of terpineol (Trans., 1906, 89, 851), namely, $d_{20^{\circ}/20^{\circ}} = 0.9385$.

Refractive power $\frac{\mu-1}{d}$ at 18.4° : $\alpha = 78.885$; $\beta = 80.466$; $\gamma = 81.450$.

Dispersion $\gamma - \alpha = 2.565$.

$$\frac{\mu^2-1}{\mu^2+2} \cdot \frac{p}{d} = 46.68 \text{ for } \alpha.$$

The *nitrosochloride*, $C_{11}H_{18}O, NOCl$.—This derivative was prepared by dissolving dihydroisocarvestrenol (2 c.c.) in ethyl nitrite (10 c.c. of 10 per cent.), and gradually adding concentrated hydrochloric acid (2 c.c.) to the solution, cooled to -10° . After standing for an hour in ice and salt, a further quantity of ethyl nitrite (10 c.c.) and hydrochloric acid (2 c.c.) was added, and the mixture allowed to remain at the ordinary temperature, when the nitrosochloride soon began to crystallise. The crystals were collected, washed with methyl alcohol, and analysed:

0.1406 gave 8 c.c. N_2 at 18° and 741 mm. $N = 6.4$.

$C_{10}H_{18}O_2NCl$ requires $N = 6.4$ per cent.

Dihydroisocarvestrenol nitrosochloride melts, with decomposition, at 125° , and is sparingly soluble in alcohol, benzene, ethyl acetate, or methyl ethyl ketone.

Formation of Derivatives of Carvestrene from Dihydroisocarvestrenol.

Carvestrene dihydrochloride, $C_{10}H_{16}, 2HCl$, is obtained when dihydroisocarvestrenol is shaken in a stoppered bottle with five times its volume of concentrated hydrochloric acid. The crystals, which will have formed after about half an hour, are collected, washed, and left in contact with porous porcelain until quite free from oil.

The colourless mass is then crystallised from methyl alcohol, from which the hydrochloride separates in glistening needles:

0.1620 gave 0.2210 $AgCl$. $Cl = 33.8$.

$C_{10}H_{18}Cl_2$ requires $Cl = 33.9$ per cent.

This hydrochloride melted sharply at 52.5° , and, when mixed with a specimen of carvestrene dihydrochloride (Trans., 1907, 91, 500), there was no alteration in melting point. When dihydrosocarvestrenol was shaken with eight volumes of hydrobromic acid (saturated at 0°) for two days, an oil still floated on the surface, but, on pouring into water and shaking vigorously, the heavy oil soon crystallised. The mass was well washed, left in contact with porous porcelain until quite colourless, and purified by crystallisation from methyl alcohol, from which the hydrobromide separated in hard, glistening, prismatic needles of melting point $48-49^{\circ}$:

0.2165 gave 0.2720 AgBr. Br = 53.5

$C_{10}H_{18}Br_2$ requires Br = 53.7 per cent.

That this substance is carvestrene dihydrobromide was proved by mixing it with a specimen of this substance which had been previously obtained (Trans., 1907, 91, 500), when there was no alteration in melting point.

cis-Tetrahydrocarvestrenediol, $C_{10}H_{18}(OH)_2$.—This derivative of carvestrene was obtained by shaking dihydrosocarvestrenol (5 c.c.) with water (600 c.c.) and sulphuric acid (10 c.c.) on the machine.

The oil was almost insoluble at first, and was therefore only slowly attacked by the dilute sulphuric acid, but, after shaking for seven days, nearly all of it had passed into solution. The product was filtered, the clear filtrate saturated with ammonium sulphate, and the cloudy liquid repeatedly extracted with ether. The ethereal solution was dried and evaporated, when a viscid syrup remained, which soon commenced to crystallise and, especially when vigorously stirred, gradually became almost solid. The mass was left in contact with porous porcelain until quite hard, and then repeatedly crystallised from ether, from which woolly masses separated. *

For analysis, the substance was dried over phosphoric oxide:

0.1223 gave 0.3120 CO_2 and 0.1280 H_2O . C = 69.6; H = 11.6.

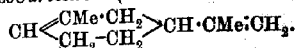
$C_{10}H_{20}O_2$ requires C = 69.8; H = 11.6 per cent.

The melting point of the *cis*-tetrahydrocarvestrenediol obtained in this experiment was 94° , or rather higher than the melting point (about 90°) found for the original specimen (Trans., 1907, 91, 501).

When, however, this original specimen was twice crystallised from ether, the melting point rose to $93-94^{\circ}$, and its identity with the substance, obtained as described above, was proved by the fact that there was no alteration in melting point when the two specimens were mixed. Careful examination of the ethereal mother liquors and of the porous porcelain used in the purification of the *cis*-tetrahydrocarvestrenediol failed to reveal even traces of the *trans*-modification,

and it was evident that the conversion of dihydroisocarvestrenol into *cis*-tetrahydrocarvestrenediol, under the above conditions, was almost quantitative.

isoCarvestrene ($\Delta^6:8^{10}$ -*m*-Menthatriene),



The conversion of dihydroisocarvestrenol into *isocarvestrene* was carried out by gradually adding the former (21 grams) to an ethereal solution of magnesium methyl iodide (containing 15 grams of magnesium), and allowing the mixture to remain for four days. The product was then decomposed by water and dilute hydrochloric acid, the ethereal solution separated, washed with water containing a little sulphurous acid, and distilled under reduced pressure. The oil was thus readily separated into two fractions: (a) boiling below 140°/200 mm., and (b) boiling at 190—200°/20 mm. When the former was repeatedly distilled over sodium under the ordinary pressure, a large fraction (11 grams) was obtained, distilling constantly at 176—177°/765 mm.:

0.1118 gave 0.3610 CO₂ and 0.1190 H₂O. C = 88.1; H = 11.8.

0.1095 „ 0.3525 CO₂ „ 0.1143 H₂O. C = 88.0; H = 11.6.

C₁₀H₁₆ requires C = 88.2; H = 11.8 per cent.

isoCarvestrene has a very pungent odour of lemons, and is oxidised slowly in the air, since a small quantity, left in a tube over water, had absorbed one-fifth of the volume of air in seven days.

When a drop of sulphuric acid is added to its solution in acetic anhydride, a violet coloration is produced, which rapidly fades.

The density determinations gave:

$$d_{20^\circ/20} = 0.8496; d_{20^\circ/4^\circ} = 0.8481.$$

Refractive power at 20°:

	μ .	$\frac{\mu-1}{d}$.	$\frac{\mu-1}{d} p$.	$\frac{\mu^2-1}{\mu^2+2} \cdot \frac{p}{d}$.
α	1.47799	0.56360	76.649	45.39
β	1.49090	0.57881	78.718	46.43
γ	1.49893	0.58829	80.007	47.09

Dispersion $\gamma - \alpha = 3.358$.

These numbers, with the exception of the density, are distinctly higher than those found for dipentene (Trans., 1906, 89, 851), namely,

$d_{20^\circ/20^\circ} = 0.8517$. Refractive power $\frac{\mu-1}{d} p$ at 14°: $\alpha = 75.602$; $\beta =$

77.391; $\gamma = 78.564$. Dispersion $\gamma - \alpha = 2.962$.

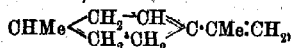
$$\frac{\mu^2-1}{\mu^2+2} \frac{p}{d} = 44.81 \text{ for } \alpha.$$

The most remarkable feature in connexion with the refraction

values for *isocarvestrene* is the fact that they are even higher than those of $dl-\Delta^3:8(9)-p$ -menthadiene (compare Trans., 1906, 89, 850), which has the values $\alpha=76.31$; $\beta=78.21$; $\gamma=79.37$;

$$\frac{\mu^2-1}{\mu^2+2} \frac{p}{d} = 45.31.$$

But $dl-\Delta^3:8(9)-p$ -menthadiene,



contains a conjugated system of two double linkings, and it has been shown (*loc. cit.*, p. 856) that this system causes a small, but distinct, rise in the values for the refraction above the normal:

	$\frac{\mu^2-1}{d} \frac{p}{d}$	Difference.
$dl-\Delta^3:8(9)-p$ -Menthadiene	76.31	
Dipentene	75.60	+0.71

The comparison of *isocarvestrene* with dipentene:

	$\frac{\mu^2-1}{d} \frac{p}{d}$	Difference.
<i>iso</i> Carvestrene	76.65	
Dipentene	75.60	+1.05

yields an even greater difference, and, as it seems impossible that elimination of water from dihydroisocarvestrenol can lead to a hydrocarbon with conjugated double linkings, it is very difficult to understand the significance of these numbers. In connexion with this point, it has been shown (Perkin, Pickles, and Tattersall, Trans., 1905, 87, 641; also 1077 and 1101) that hydrocarbons containing a conjugated system of double linkings exhibit a characteristic behaviour when treated with bromine, hydrogen bromide, or hydrogen chloride. Thus, for example, $dl-\Delta^3:8(9)-p$ -menthadiene combines with one molecule of each of these, whereas dipentene, which does not contain conjugated double linkings, combines, as is well known, with two molecules in each case. It is very remarkable that *isocarvestrene* appears to behave in this matter in a similar way to $dl-\Delta^3:8(9)-p$ -menthadiene. When *isocarvestrene* (1.25 grams), dissolved in three times its volume of dry chloroform and cooled to -10° , was titrated with bromine, a permanent straw-colour was produced when 1.7 grams of the halogen had been added, but the end point was not very sharp. This result agrees best with the formation of a dibromide, $\text{C}_{10}\text{H}_{16}\text{Br}_2$, which requires the addition of 1.47 grams of bromine, whereas the quantity required for the formation of a tetrabromide, $\text{C}_{10}\text{H}_{16}\text{Br}_4$, is 2.95 grams. *isocarvestrene* did not dissolve when it was shaken in a sealed tube with 5 volumes of a solution of hydrogen bromide in acetic acid saturated at 0° ; the solution became brown, but the hydrocarbon was not converted into a crystalline dihydrobromide, $\text{C}_{10}\text{H}_{18}, 2\text{HBr}$.

When, after several days, the product was poured into water, a rather heavy oil separated, which showed no tendency to crystallise. Carvestrene was then dissolved in dry ether, cooled to -10° , and saturated with hydrogen chloride; the solution soon became brown and then a deep claret, and yielded a resinous mass when it was washed with water and evaporated.

Again, when the hydrocarbon was shaken for ten days with ten times its volume of concentrated hydrochloric acid, no crystalline dihydrochloride separated. The product was poured into water, and the oil, after extraction with ether in the usual manner, analysed and found to contain 14.7 per cent. of chlorine, which is less even than that required for the mono-hydrochloride, $C_{10}H_{16}HCl$, namely, 20.6 per cent.

It has been pointed out on p. 1888 that dihydroisocarvestrenol readily combines with halogen acids to yield the carvestrene derivatives $C_{10}H_{16} \cdot 2HCl$ and $C_{10}H_{16} \cdot 2HBr$, and it is difficult to understand why isocarvestrene does not show the same behaviour, especially when it is remembered that carvestrene, under the same conditions, combines readily with halogen acids (compare Trans., 1907, 91, 500).

During the fractionation of the product of the action of magnesium methyl iodide on dihydroisocarvestrenol (p. 1890), a considerable quantity of an oil was obtained, distilling at $190-200^{\circ}/20$ mm.

When this was carefully fractionated over sodium, it boiled very constantly at $188-190^{\circ}/20$ mm., and yielded the following analysis:

0.1560 gave 0.5050 CO_2 and 0.1620 H_2O . $C = 88.2$; $H = 11.6$.

$C_{20}H_{32}$ requires $C = 88.2$; $H = 11.8$.

The determination of the molecular weight by the cryoscopic method in benzene solution gave $M.W. = 254$, whereas the formula $C_{20}H_{32}$ requires $M.W. = 272$. This diterpene, for which we propose the name *disocarvestrene*, is a viscid, colourless syrup, which has a faint odour of peppermint, and develops a pink coloration when a drop of sulphuric acid is added to its solution or suspension in acetic anhydride. If a drop of bromine is added to a solution of the diterpene in chloroform at -10° , a straw-colour is at once produced, and, on standing at the ordinary temperature, the solution soon becomes deep purple and, after twelve hours, an intense indigo-blue.

During the course of other investigations of this series, diterpenes similar to disocarvestrene have been isolated, on several occasions, from the products of the dehydration of some of the corresponding alcohols. It seems possible that, during their formation, the characteristic group, $-CMe:CH_2$, of two molecules of the simple terpene

undergoes polymerisation with formation of a tetramethylene derivative containing the group



We take this opportunity of thanking Mr. W. N. Haworth for valuable assistance during this difficult investigation, and we wish also to express our thanks to the Research Fund Committees of the Royal Society and Chemical Society for grants which have covered much of the expense involved.

THE SCHUNCK LABORATORY,
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CLXXXIX.—*Aromatic Arsonic Acids.*

By MARMADUKE BARROWCLIFF, FRANK LEE PYMAN, and FREDERIC GEORGE PERCY REMFRY.

In a previous communication from these laboratories (Trans., 1908, 93, 1180), some account was given of the preparation and properties of certain aromatic arsonic and arsinic acids, and reference was made to the use of substances of this class in trypanosomiasis. At that time, the physiological results obtained with the bis-aminoarylarsinic acids were not sufficiently conclusive for publication, but Dr. Breinl has now found that sodium bis-2-aminotolyl-5-arsinate and sodium bis-2-acetylaminotolyl-5-arsinate are of little use in trypanosomiasis, probably owing to their low arsenic content.

The present communication records the chemical and physical properties of a number of new arsonic acids, which are derivatives of *p*-aminophenylarsonic acid and 2-aminotolyl-5-arsonic acid. These two acids have, of course, a certain toxicity, although this is only a small fraction of that of arsenic acid, and it was thought at one time that they were slowly decomposed in the body with formation of arsenic acid and aniline or *o*-toluidine respectively, and that their toxicity was due partly to the arsenic acid and partly to the aniline. We therefore prepared *p*-hydroxyphenylarsonic acid and 2-hydroxytolylarsonic acid by diazotising the corresponding amino-acids and removing the nitrogen, in order that when the arsenic acid was decomposed in the body, phenol and *o*-cresol should result instead of

aniline and *o*-toluidine. Physiological investigation of the sodium salts of these acids, however, has proved that they are of no use in trypanosomiasis, behaving in the body like arsenic acid rather than like *p*-aminophenylarsonic acid. The reason for the difference in action between the amino- and hydroxy-arylarsonic acids has now been supplied by Nierenstein (*Annals of Tropical Medicine and Parasitology*, 1908, 2, 249), who has shown that sodium *p*-aminophenylarsonate and its monoacyl derivatives combine with proteins, whilst its diacyl derivatives, sodium *p*-hydroxyphenylarsonate, and sodium arsenate do not.

During the period of the physiological investigation of these substances, a part of our chemical results has been anticipated, for Berthelm (*Ber.*, 1908, 41, 1854) described the preparation and properties of sodium *p*-hydroxyphenylarsonate, although he did not isolate the free acid, and Benda and Kahn (*Ber.*, 1908, 41, 1678) described the preparation and properties of 2-hydroxytolyl-5-arsonic acid; consequently, we have only recorded here the preparation and properties of salts and derivatives which these chemists have not described.

The trypanocidal action of certain benzidine and triphenylmethane dyes led us to inquire whether dyestuffs which contain a phenylarsonic acid residue would be more effective than the substituted phenylarsonic acids themselves. In order to test this point, we have prepared certain azobenzene-*arsonic* acids and phenazine-*arsonic* acids.

The production of a red dyestuff on diazotising *p*-aminophenylarsonic acid and coupling with β -naphthylamine has already been described by Ehrlich and Berthelm (*Ber.*, 1907, 40, 3297), but no results of any physiological examination of this substance have as yet been available. By diazotising *p*-aminophenylarsonic acid and 2-aminotolyl-5-arsonic acid, and coupling with phenol, β -naphthol, and dimethylaniline respectively, we have prepared 4-hydroxyazobenzene-4'-arsonic acid, β -naphtholazobenzene-4'-arsonic acid, 4-dimethylaminoazobenzene-4'-arsonic acid, 4-hydroxy-2'-methylazobenzene-4'-arsonic acid, and 4-dimethylamino-2'-methylazobenzene-4'-arsonic acid. Of these, the two last were physiologically examined, and proved to be of very small value against a virulent strain of trypanosomes.

The preparation of a phenazine from *p*-aminophenylarsonic acid by the method of Perkin and Nierenstein (*Trans.*, 1905, 87, 1412) was suggested to us by Dr. Nierenstein, and this has been carried out by the formation of phenazine-2:7-bis-arsonic acid; this, however, proved to have little effect on trypanosomes. The corresponding phenazine from 2-aminotolyl-5-arsonic acid, 4:9-dimethylphenazine-2:7-bis-arsonic acid, was also prepared, but, in view of the inactivity of the lower homologue, it was not submitted to physiological examination.

The physiological examination of these substances was carried out at the Runcorn Research Laboratories of the Liverpool School of Tropical Medicine, and we take this opportunity of expressing our thanks to Dr. Breinl, the Director of these Laboratories.

EXPERIMENTAL.

p-Hydroxyphenylarsonic Acid, $\text{HO}\cdot\text{C}_6\text{H}_4\cdot\text{AsO}(\text{OH})_2$.

Two hundred and fifty grams of sodium *p*-aminophenylarsonate (containing $5\text{H}_2\text{O}$) were diazotised with the calculated quantities of sodium nitrite and hydrochloric acid, and the solution evaporated to dryness. The residue was then ground to a fine powder, and again heated on the water-bath until completely dry, when the *p*-hydroxyphenylarsonic acid was separated from the sodium chloride by dissolution in boiling acetone, from which it crystallised on cooling in salmon-pink, prismatic needles. The yield amounted to 125 grams, that is, 75 per cent. of the theoretical.

p-Hydroxyphenylarsonic acid is obtained almost colourless by several crystallisations from acetone, but persistently retains a faint pink tinge. It melts at $170\text{--}174^\circ$, and is very easily soluble in water or alcohol, but sparingly so in cold acetone:

0.1724 gave 0.2110 CO_2 and 0.0508 H_2O . $\text{C} = 33.4$; $\text{H} = 3.3$.

$\text{C}_6\text{H}_5\text{O}_4\text{As}$ requires $\text{C} = 33.0$; $\text{H} = 3.2$ per cent.

p-Acetoxyphenylarsonic Acid, $\text{CH}_3\cdot\text{CO}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{AsO}(\text{OH})_2$.

Twelve grams of *p*-hydroxyphenylarsonic acid were gently boiled for one hour with 36 grams of acetic anhydride and 0.5 gram of concentrated sulphuric acid. The whole of the arsonic acid first dissolved, and then separated as a jelly, which was ground with etheroleum several times to remove the acetic anhydride, and re-crystallised from acetone, giving an almost quantitative yield of the acetyl derivative.

p-Acetoxyphenylarsonic acid crystallises from acetone in matted clusters of fine needles, which do not melt at 250° . It is very easily soluble in cold water, but somewhat sparingly so in cold alcohol or acetone:

0.2092 gave 0.2822 CO_2 and 0.0650 H_2O . $\text{C} = 36.8$; $\text{H} = 3.5$.

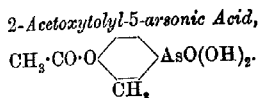
$\text{C}_8\text{H}_7\text{O}_6\text{As}$ requires $\text{C} = 36.9$; $\text{H} = 3.5$ per cent.

The sodium salt crystallises from water in matted clusters of fine needles, and contains 3 molecules of water of crystallisation. It is very readily soluble in water, giving a neutral solution, but sparingly so in alcohol:

0.3830, air-dried salt, lost 0.0600 at 100°. $H_2O = 15.7$.
 $C_8H_8O_5AsNa, 3H_2O$ requires $H_2O = 16.1$ per cent.

Sodium 2-hydroxytolyl-5-arsenate crystallises from water in plates, which contain two molecules of water of crystallisation. It is soluble in four times its weight of cold water, giving a neutral solution, and is sparingly soluble in alcohol:

0.4230, air-dried salt, lost 0.0528 at 100°. $H_2O = 12.5$.
 $C_7H_8O_4AsNa, 2H_2O$ requires $H_2O = 12.4$ per cent.

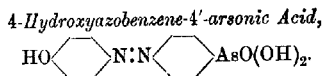


This acid is prepared from 2-hydroxytolyl-5-arsonic acid in a manner analogous to the preparation of *p*-acetoxyphenylarsonic acid. It separates from acetone in clusters of fine needles, and melts at 164—166°; it is sparingly soluble in cold water or acetone, but readily so in alcohol:

0.1684 gave 0.2438 CO_2 and 0.0600 H_2O . $C = 39.5$; $H = 4.0$.
 $C_9H_{11}O_5As$ requires $C = 39.4$; $H = 4.1$ per cent.

The *sodium* salt crystallises from water in radial clusters of silky needles, and contains 4 molecules of water of crystallisation. It is readily soluble in water, giving a neutral solution, but sparingly so in alcohol:

0.2594, air-dried salt, lost 0.0498 at 100°. $H_2O = 19.2$.
 $C_9H_{10}O_6AsNa, 4H_2O$ requires $H_2O = 19.6$ per cent.



Forty-five grams of sodium *p*-aminophenylarsonate (containing $5H_2O$) were dissolved in 100 c.c. of water and 40 grams of hydrochloric acid, and diazotised at 0° with an aqueous solution of sodium nitrite until the mixture contained free nitrous acid. A cold solution of 13 grams of phenol in 125 grams of 10 per cent. aqueous sodium hydroxide was then added, followed by a sufficient further quantity of aqueous sodium hydroxide to form a clear solution. After standing for one hour, the deep red solution was acidified with hydrochloric acid, and the dye collected. In order to purify it, it was treated with sufficient 10 per cent. aqueous sodium hydroxide to form the disodium salt, warmed, and saturated with carbon dioxide; on cooling,

the monosodium salt separated in glistening, yellow plates; these were collected, re-dissolved in hot water, and acidified with acetic acid:

0.1193 gave 0.1971 CO_2 and 0.0373 H_2O . $\text{C} = 45.0$; $\text{H} = 3.5$.

$\text{C}_{12}\text{H}_{11}\text{O}_4\text{N}_2\text{As}$ requires $\text{C} = 44.7$; $\text{H} = 3.4$.

4-Hydroxyazobenzene-4'-arsonic acid is a light red powder, and is insoluble in water and all the usual organic solvents. It is readily soluble in alkalis, giving deep red solutions.

The monosodium salt, prepared as above, is almost insoluble in cold water, and sparingly so in hot water. It contains $2\frac{1}{2}$ molecular proportions of water of crystallisation:

0.2816, air-dried salt, lost 0.0322 at 115° . $\text{H}_2\text{O} = 11.4$.

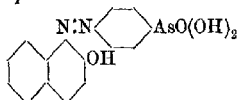
$\text{C}_{12}\text{H}_{10}\text{O}_4\text{N}_2\text{AsNa}, 2\frac{1}{2}\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 11.6$ per cent.

The disodium salt is prepared by dissolving the monosodium salt in one molecular equivalent of aqueous sodium hydroxide, concentrating, and mixing with alcohol, when it is obtained as a light red powder, which is readily soluble in cold water. It contains 8 molecules of water of crystallisation:

0.4576 lost 0.1268 at 115° . $\text{H}_2\text{O} = 27.7$ per cent.

$\text{C}_{12}\text{H}_8\text{O}_4\text{N}_2\text{AsNa}_2, 8\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 28.2$ per cent.

β -Naphtholazobenzene-4'-arsonic Acid,



A diazotised solution of *p*-aminophenylarsonic acid was rendered alkaline, and coupled with an alkaline solution of one molecular equivalent of β -naphthol. The resulting acid was precipitated on the addition of hydrochloric acid. It was re-dissolved in sufficient aqueous sodium hydroxide to form a disodium salt, and a stream of carbon dioxide was passed through the solution, when the monosodium salt was obtained as a deep orange, crystalline precipitate. The acid was regenerated from this and analysed:

0.1702 gave 0.3212 CO_2 and 0.0582 H_2O . $\text{C} = 51.5$; $\text{H} = 3.8$.

$\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}_2\text{As}$ requires $\text{C} = 51.6$; $\text{H} = 3.5$ per cent.

β -Naphtholazobenzene-4'-arsonic acid is a bright red powder, which is soluble only in alkalis.

The monosodium salt, prepared as above, is sparingly soluble in boiling water; it contains 5 molecules of water of crystallisation:

0.3526, air-dried salt, lost 0.0660 at 115° . $\text{H}_2\text{O} = 18.7$.

$\text{C}_{16}\text{H}_{12}\text{O}_4\text{N}_2\text{AsNa}, 5\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 18.6$ per cent.

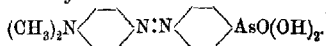
The disodium salt is a dark red powder, which is readily soluble in

water, and contains $6\frac{1}{2}$ molecular proportions of water of crystallisation :

0.3692 lost 0.0823 H_2O . $H_2O = 22.3$.

$C_{10}H_{11}O_4N_2AsNa_2 \cdot 6\frac{1}{2}H_2O$ requires $H_2O = 22.0$ per cent.

4-Dimethylaminoazobenzene-4'-arsonic Acid,



Forty-five grams of sodium paminophenylarsonate were dissolved in 100 c.c. of water and 40 grams of concentrated hydrochloric acid, and diazotised at 0° with 9.5 grams of sodium nitrite dissolved in 50 c.c. of water. 16.5 Grams of dimethylaniline dissolved in 15 grams of hydrochloric acid and 150 c.c. of water were then added, and, after several hours, the dye was precipitated by the addition of an excess of sodium acetate solution, collected, washed with water, and dried. For analysis, the acid was regenerated from its recrystallised monosodium salt, washed, and dried :

0.1473 gave 0.2584 CO_2 and 0.0580 H_2O . $C = 47.8$; $H = 4.4$.

$C_{14}H_{16}O_3N_2As$ requires $C = 48.1$; $H = 4.6$ per cent.

4-Dimethylaminoazobenzene-4'-arsonic acid is a red powder, which is insoluble in water and the usual solvents, but readily soluble in alkalis and mineral acids. The acid salts are decomposed by water.

The monosodium salt crystallises from hot water in glistening, scarlet plates, which are sparingly soluble in cold water, and contain $5\frac{1}{2}$ molecular proportions of water of crystallisation :

0.2398 lost 0.0506 at 115° . $H_2O = 21.1$.

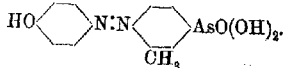
$C_{14}H_{15}O_3N_2AsNa \cdot 5\frac{1}{2}H_2O$ requires $H_2O = 21.1$ per cent.

The disodium salt was prepared by dissolving the monosodium salt in the calculated quantity of aqueous sodium hydroxide, concentrating to a small bulk, and precipitating with alcohol. It is a scarlet powder, which is soluble in 3 parts of cold water, and contains 6 molecules of water of crystallisation :

0.3644 lost 0.0756 at 115° . $H_2O = 20.7$.

$C_{14}H_{14}O_3N_2AsNa_2 \cdot 6H_2O$ requires $H_2O = 21.4$ per cent.

4-Hydroxy-2'-benzeneazotoluene-5'-arsonic Acid,



Fifty grams of sodium 2-aminotolyl-5-arsonate were diazotised and coupled with an alkaline solution of phenol, as in the preparation of 4-hydroxyazobenzene-4'-arsonic acid. In this case, however, the clear

alkaline solution was treated with carbon dioxide, when a red, crystalline precipitate, consisting of the monosodium salt, was formed, the yield amounting to 84 per cent. of the theoretical. A portion of the salt was recrystallised, dissolved in hot water, and acidified with acetic acid, when a crystalline precipitate of the arsonic acid was obtained:

0.1412 gave 0.2410 CO_2 and 0.0500 H_2O . $\text{C} = 46.5$; $\text{H} = 3.9$,

$\text{C}_{13}\text{H}_{11}\text{O}_4\text{N}_2\text{As}$ requires $\text{C} = 46.4$; $\text{H} = 3.9$ per cent.

4-Hydroxy-2'-benzeneazotoluene-5'-arsonic acid is a light red, crystalline powder, which is practically insoluble in boiling water and the usual organic solvents, but readily soluble in alkalis.

The monosodium salt crystallises from water in red leaflets. It is moderately soluble in hot water, sparingly so in cold, and contains $2\frac{1}{2}$ molecular proportions of water of crystallisation:

0.5797 lost 0.0655 at 115° . $\text{H}_2\text{O} = 11.3$.

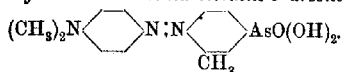
$\text{C}_{13}\text{H}_{12}\text{O}_4\text{N}_2\text{As}, 2\frac{1}{2}\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 11.1$ per cent.

The disodium salt was prepared by dissolving the monosodium salt in the calculated quantity of aqueous sodium hydroxide, concentrating to a small bulk, and adding alcohol. It is a red powder soluble in 3 parts of cold water, and contains $4\frac{1}{2}$ molecular proportions of water of crystallisation:

0.6185 lost 0.1081 at 115° . $\text{H}_2\text{O} = 17.5$.

$\text{C}_{13}\text{H}_{11}\text{O}_4\text{N}_2\text{AsNa}_2, 4\frac{1}{2}\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 17.6$ per cent.

4-Dimethylamino-2'-benzeneazotoluene-5'-arsonic Acid,



Forty-five grams of sodium 2-aminotolyl-5-arsonate were dissolved in dilute hydrochloric acid, diazotised, and coupled with an acid solution of dimethylaniline. The solution was allowed to stand for an hour and neutralised with sodium carbonate, and the resulting solid was collected, dissolved in sodium hydroxide, and precipitated as the crystalline monosodium salt by carbon dioxide, the yield amounting to 82 per cent. of the theoretical. The acid was obtained from the monosodium salt, by acidifying the hot aqueous solution with acetic acid, as a red, crystalline powder, which formed salts with mineral acids and with bases:

0.1456 gave 0.2642 CO_2 and 0.0630 H_2O . $\text{C} = 49.5$; $\text{H} = 4.8$.

$\text{C}_{15}\text{H}_{18}\text{O}_3\text{N}_3\text{As}$ requires $\text{C} = 49.6$; $\text{H} = 4.9$ per cent.

The monosodium salt is a red, crystalline, powder which is moderately

easily soluble in hot water, but sparingly so in cold. It contains 5 molecules of water of crystallisation:

0.4418 lost 0.0840 at 115°. $H_2O = 19.0$.

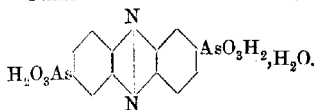
$C_{15}H_{17}O_3N_3AsNa, 5H_2O$ requires $H_2O = 18.9$ per cent.

The *disodium* salt, prepared in the usual manner, is a red powder, which is soluble in 3 parts of cold water, and contains 4 molecules of water of crystallisation:

0.4250 lost 0.0628 at 115°. $H_2O = 14.8$.

$C_{15}H_{16}O_3N_3AsNa_2, 4H_2O$ requires $H_2O = 15.0$ per cent.

Phenazine-2:7-bis-arsonic Acid,



Twenty grams of *p*-aminophenylarsonic acid were suspended in 100 c.c. of water, and dissolved by the addition of 15 c.c. of concentrated sulphuric acid. To the warm solution, 23 grams of finely powdered ammonium persulphate were added in small quantities during an hour with thorough stirring. The light brown liquor was then gently heated on the water-bath, when it darkened in colour, and, after about ten minutes, a brisk evolution of gas commenced and small, leafy crystals were deposited. The solution was removed from the water-bath until the action had moderated, then replaced for a further five minutes, and finally allowed to cool for one and a-half hours. The light brown, leafy crystals were collected, well washed, and air-dried; the yield was 7.2 grams, that is, 35 per cent. of the theoretical. The acid was purified by converting it into the sodium salt, crystallising this, and regenerating.

Phenazine-2:7-bis-arsonic acid does not melt at 300°. It is insoluble in water, very sparingly soluble in alcohol or acetic acid, and insoluble in the other usual organic solvents. It gives a blood-red coloration with concentrated sulphuric acid. It contains one molecule of water of crystallisation, which is not lost either by standing in a vacuum desiccator or on heating at 120°. At 150°, however, the acid loses 2 molecules of water, and must therefore form an internal anhydride.*

0.2112, after drying in a vacuum desiccator, gave 0.2524 CO_2 and

0.0518 H_2O . $C = 32.6$; $H = 2.7$.

0.2470 gave 13.4 c.c. N_2 at 17° and 769 mm. $N = 6.3$.

* *p*-Aminophenylarsonic acid loses one molecule of water at 150°, giving an anhydride, $NH_2 \cdot C_6H_4 \cdot AsO_2$.

0.2609 lost 0.0205 at 150°. $H_2O = 7.9$.
 $C_{13}H_{10}O_6N_2As_2$, H_2O requires $C = 32.3$; $H = 2.7$; $N = 6.4$; and loss of
 $2H_2O = 8.5$ per cent.

0.1830, dried at 150°, gave 0.2358 CO_2 and 0.0362 H_2O . $C = 35.1$;
 $H = 2.2$.

$C_{13}H_8O_6N_2As_2$ requires $C = 35.1$; $H = 2.0$ per cent.

Sodium salt was prepared by suspending the acid in water,
 and adding aqueous sodium hydroxide to dissolve it:

Required 0.0943 NaOH, that is, one mol. acid required
 4.1 mols. NaOH.

On adding the aqueous solution to small bulk and adding
 alcohol, a buff-coloured crystalline powder,
 which contains 11 molecules of water of crystallisation. It is soluble
 in one and a-half times its weight of water at 20°, giving an alkaline
 solution, and is insoluble in alcohol:

0.5229, air-dried salt, lost 0.1462 at 100°. $H_2O = 28.0$.

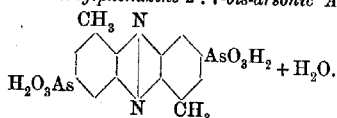
$C_{12}H_6O_6N_2As_2Na_4$, 11 H_2O requires $H_2O = 27.8$ per cent. *

When this salt is allowed to stand in a vacuum desiccator, it loses
 1½ molecules of water, and changes colour, becoming bright red.
 After removing the residual water of crystallisation at 100°, the
 substance again becomes buff-coloured:

0.5736, desiccator-dried salt, lost 0.1435 at 100°. $H_2O = 25.0$.

$C_{12}H_6O_6N_2As_2Na_4$, 9½ H_2O requires $H_2O = 24.9$ per cent.

4 : 9-Dimethylphenazine-2 : 7-bis-arsonic Acid,



This acid was prepared by the oxidation of 2-aminotolyl-5-arsonic
 acid in a manner similar to the preparation of phenazine-2:7-bis-arsonic
 acid. The yield, however, was smaller, and the acid was obtained in
 the form of a fine, buff-coloured powder, which did not melt at 300°. It
 is insoluble in water, very sparingly soluble in alcohol and acetic
 acid, and insoluble in the other usual organic solvents. It gives a
 blood-red coloration with concentrated sulphuric acid:

0.1287 gave 0.1682 CO_2 and 0.0393 H_2O . $C = 35.6$; $H = 3.4$.

$C_{14}H_{14}O_6N_2As_2$, H_2O requires $C = 35.4$; $H = 3.4$ per cent.

THE WELLCOME CHEMICAL WORKS,
 DARTFORD, KENT.

CXC.—*The Relation between Absorption Spectra and Chemical Constitution. Part XI. Some Aromatic Hydrocarbons.*

By EDWARD CHARLES CYRIL BALY and WILLIAM BRADSHAW TUCK

IN an investigation on the absorption spectra of a number of derivatives of naphthalene, undertaken with the view of determining the influence of substitution, we have made a careful study of the absorption spectrum of naphthalene. Realising the great importance of this spectrum, we have taken every precaution as regards the purity of the naphthalene, and have used more than one source of light in order to guard against any error arising from inequalities in the spectrum of the source.

The purest naphthalene was obtained from Kahlbaum, and this was heated with manganese dioxide and concentrated sulphuric acid, after which it was distilled in a current of steam. This process was repeated, and then the naphthalene was converted into the picrate, this compound being carefully recrystallised. The naphthalene, after regeneration, was again distilled in a current of steam, and then recrystallised from dilute alcohol. The absorption curve we obtained with the pure compound is shown in Fig. 1 (full curve); there are three absorption bands, namely, two narrow ones at $1/\lambda = 3125$ and 3220 respectively, and a third broad band with its head at about $1/\lambda = 3700$. Our curve differs from that given by Hartley (Trans., 1885, 47, 685) in not showing bands at $1/\lambda = 3270$ and 3370 .

The absorption of naphthalene is of peculiar interest, since owing to the structure of the compound, two ethylenic linkings are conjugated with a benzene nucleus, the whole forming one closed system. Among the substituted aromatic compounds which have been dealt with up to the present, there have only been included those in which the unsaturated centres are attached to the benzene nucleus at one point. It would appear from previous observations that the absorption spectrum of a benzene compound containing a side-chain with ethylenic double linkings shows one broad absorption band. A good example of this is stilbene, the absorption curve of which is shown in Fig. 4 (dotted curve). We are therefore able to say that when we have centres of residual affinity, as expressed by ethylenic linkings attached at one point to a benzene ring, the resulting isorropesis gives rise to a single absorption band which is much nearer to the red than the band

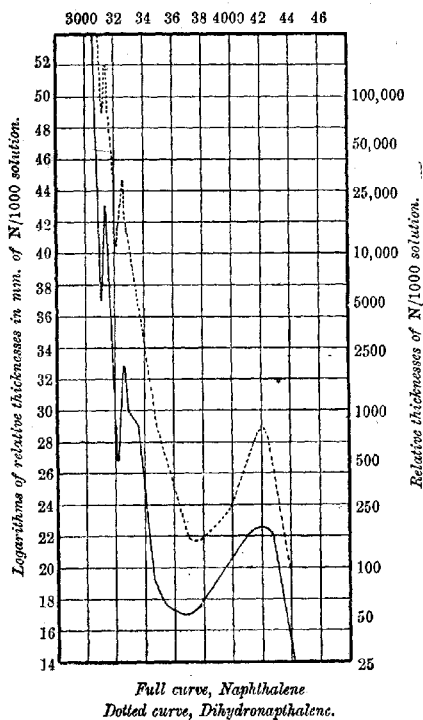
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of benzene, the amount of the shift being dependent upon the number of ethylene linkings in the side-chain.

The case is different, however, when the unsaturated system is attached at two points to the benzene ring; the absorption is of a very different type, as can be seen from the curves of

FIG. 1.

Oscillation frequencies.

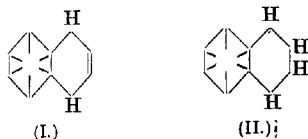


naphthalene, anthracene, and phenanthrene. In these cases, and in all the hydrocarbons examined of this type, there is always present in the absorption a band very close to the position of the benzene bands, and also small bands or groups of bands appearing nearer the red end of the spectrum and at greater concentra-

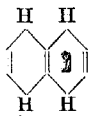
tion. In these cases, therefore, we have direct evidence that true benzenoid motions are taking place, and that also there are new free periods of vibration due to the isorropesis between the ethylenic linkings and the benzene ring. This type of oscillation is due in some way to the fact that the unsaturated side-chain is doubly linked to the phenyl-group, for if there be only a single point of attachment the system acts as a whole, giving only one free period of vibration.

In reference to this linking together of the systems, it is interesting to compare the absorption of hexatriene, $\text{CH}_2\text{:CH}\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}_2$, with that of benzene. The absorption of the former is shown in Fig. 4 (dot and dash curve), and, as can be seen, only shows general absorption, whereas in benzene, which contains the same number of ethylenic linkings, we have a complicated system of absorption bands due to the play of forces which become possible owing to the formation of the ring.

It is necessary, in forming any views on the play of forces in naphthalene, to consider the chemical behaviour of the compound. From the ease with which naphthalene is reduced in hot alcoholic solution by metallic sodium to the dihydro-compound (I),



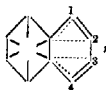
and the further reduction to the tetrahydro-compound (II) by the action of sodium on a hot solution in amyl alcohol, this being the final product of reduction (Bamberger and Lodter, *Ber.*, 1887, **20**, 3075; Bamberger and Kitschelt, *Ber.*, 1890, **23**, 1561), there is little doubt that one of the rings is truly benzenoid, and the other contains two ethylenic double bonds which, according to Thiele's law, give their maximum effect at the two extremes; that is to say, at the positions 1 and 4. Again, reduction of both rings takes place only when naphthalene is heated with phosphorus and hydriodic acid, a method which will reduce benzene itself. It is interesting to note that one of the first compounds produced by the action of phosphorus and hydriodic acid on naphthalene is the α -tetrahydro-compound,



This reduction no doubt takes place in two stages: first, the addition of an atom of hydrogen at the two extremes of the conjugated ethylenic double linkings, as in the case of the action of metallic sodium on a solution of naphthalene in alcohol; secondly, the addition of hydrogen to the top and bottom of the benzenoid ring, a reduction which would follow naturally from the motions of the benzene ring as put forward in a previous paper (Baly, Edwards, and Stewart, *Trans.*, 1906, **89**, 514), where it was shown that the maximum unsaturation occurs in benzene at two carbon atoms opposite to one another.

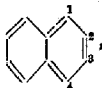
Naphthalene therefore would seem to consist of two rings, of which one is truly benzenoid, and the other contains two conjugated double linkings. There is no reason to insist that there is no possibility of an interchange of the characteristics from one ring to the other, that is, there is no reason to insist that the two rings are permanently endowed with one of the two above characters; in fact, it seems that the interchange of these characters between the rings is perfectly possible, and no doubt is continually taking place.

In seeking the origin of the three absorption bands in the spectrum of naphthalene, it may be said at once that the broad absorption band with its head at $1/\lambda = 3700$ is due to the benzenoid motions of the naphthalene molecule; we may attribute this band therefore to the half of the molecule which is benzenoid in character. The two narrow bands of $1/\lambda = 3125$ and 3220 , which are much nearer to the red than any of the benzene bands, must be due to the isorropesis between the benzenoid tautomerism of the ring and the ethylenic double linkings of the other. If the molecule of naphthalene is written



with the centric formula to represent the benzenoid ring, it is evident that the interference or isorropesis between the ethylene system and the benzenoid system can be imagined to occur either between the atoms 1, 4, and the benzenoid ring, or between the atoms 2, 3, and the benzenoid ring. In the figure this possible play of forces is denoted by the two pairs of dotted lines. Now the first alternative, namely, the isorropesis between the atoms 1, 4, and the benzenoid system, does not seem to be valid, because it is merely the first stage in the interchange of character between the two rings. If the dotted lines between the atoms 1, 4, and the

adjacent ring represent real double linkings, the molecule would become



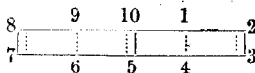
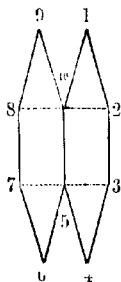
or, in other words, the ring 1:2:3:4 would now be the benzenoid ring, whilst the other would contain the ethylenic system. Inasmuch as it is necessary to have isorropesis or interference between two separate and distinct systems in order to produce bands of smaller free period than the benzene bands, so it would not seem possible to attribute either of the naphthalene bands at $1/\lambda = 3125$ and 3220 to the above process, a process which is merely a step in the interchange of character between the two naphthalene rings.

We are thus left with the second alternative, namely, the isorropesis between the atoms 2 and 3, with the benzenoid system as expressed by the dotted lines:



This is the only way in which the two systems, ethylenic and benzenoid, can give an interference system without either of them losing their character.

This may be made clearer by the conception that the naphthalene molecule is in a state of pulsation in somewhat the same way as has already been suggested for the benzene molecule (Baly, Edwards, and Stewart, *loc. cit.*):



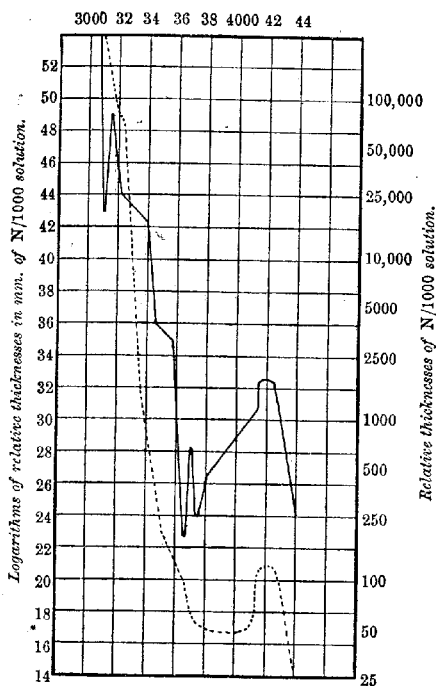
The molecule would then have two extreme phases, as shown in the figures, and the readiness with which isorropesis can arise

between the ethylenic and benzenoid systems through the atoms 2 and 3 is at once apparent.

It is evident that the two central atoms 5 and 10 are the two most important to be considered in determining the types of isomerism arising in the naphthalene molecule. In a previous

FIG. 2.

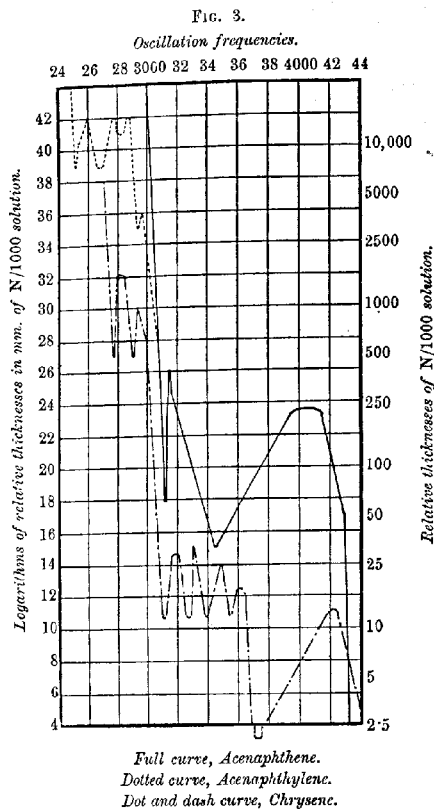
Oscillation frequencies.

Full curve, α -Tetrahydronaphthalene.

Dotted curve, Tetrahydronaphthalene.

paper (Baly and Collie, Trans., 1905, 87, 1332) on the dynamics of the benzene molecule, it was shown that each carbon atom is concerned in the making and breaking of an ortho-, meta-, and para-linking respectively. In naphthalene, however, the two carbon atoms 5 and 10 can only be concerned in making and breaking of ortho- and meta-linking respectively, for unless the rings are

distorted to an inconceivable degree, the atoms 5 and 8 cannot approach one another sufficiently near to enable a para-linking to be formed, the same being true of the atoms 10 and 7. As far as the isorropesis is concerned, only two types are possible, namely, (1) when the atoms 5 and 10 are making and breaking

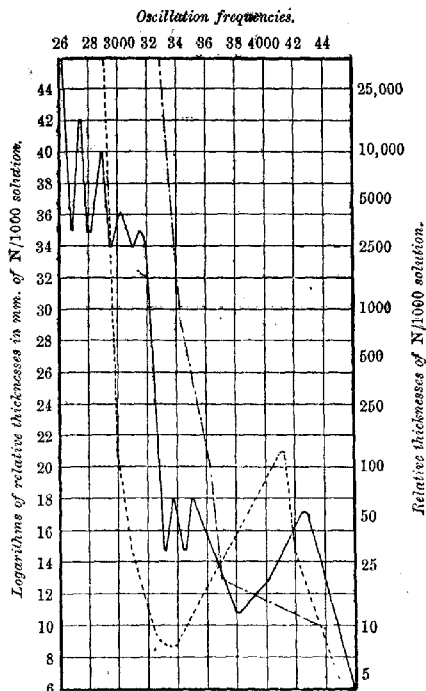


an ortho-linking; and (2) when the atoms 5 and 10 are making and breaking a meta-linking. There should, according to this view, be only two isorropesis bands in the absorption spectrum of naphthalene, and, indeed, we find only two, namely, those at $1/\lambda = 3125$ and 3220 . It is possible to put this explanation to a somewhat rigid test by examining the absorption spectra of dihydro-

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naphthalene and the two tetrahydro-compounds. If our theory is correct, it is a simple matter to forecast the absorption spectra of these compounds. Thus the tetrahydronaphthalene with one ring entirely reduced should merely show absorption due to

FIG. 4.

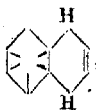


Full curve, Fluorene.

Dotted curve, Stilbene.

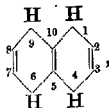
Dot and dash curve, Hexatriene.

benzenoid tautomerism of the remaining ring, without either of the isorropesis bands. The dihydronaphthalene,



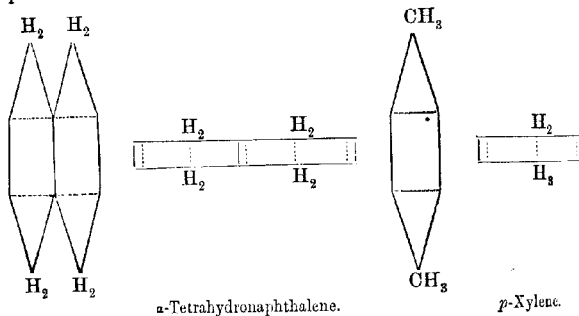
1910 BALLY AND TUCK: THE RELATION BETWEEN ABSORPTION

should exhibit an absorption very similar to that of naphthalene, since the residual affinity of the atoms 2 and 3 is only slightly reduced, so that the play of forces within the molecule is only slightly lessened in degree. The so-called α -tetrahydronaphthalene,



is peculiarly interesting, because the play of forces is rendered very much simpler. One possibility, namely, the make and break of linking between the ortho-atoms 5 and 6 and 10 and 9, has been entirely eliminated, so that we should expect one of the isorropesia bands to be removed. These three compounds have been prepared and their spectra examined, and, as can be seen on reference to Fig. 1 (dotted curve) and Fig. 2 (both curves), they conform absolutely to what the theory foretold.

A further point of interest arises in connexion with α -tetrahydronaphthalene; both rings are now exactly similar, and so the pulsating movements of the molecule should be more regular and less distorted than in naphthalene itself. In fact, the motions may be directly compared with those of *p*-xylene, and may be represented in their extreme phases as follows:



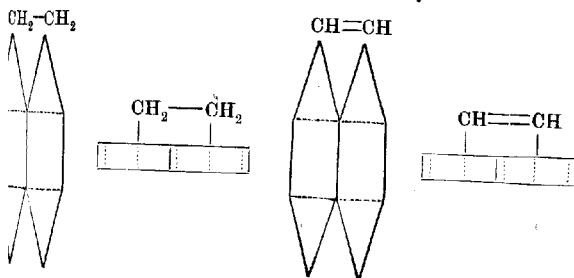
The benzenoid absorption bands of α -tetrahydronaphthalene should closely resemble those of *p*-xylene. The absorption of *p*-xylene has already been described (Bally and Ewbank, *Trans.*, 1905, **87**, 1355), and shows two absorption bands of exactly the same type and in exactly the same position as the two of α -tetrahydronaphthalene. The oscillation frequencies of the heads of the bands in each case are:

$$1/\lambda = 3650 \text{ and } 3730.$$

The three reduced naphthalenes thus afford complete support to the theory advanced above, and, moreover, enable us to determine which of the two isorropesis bands of naphthalene can be attributed to each of the two isorropic processes. The results obtained with the α -tetrahydro-compound show that the band at $1/\lambda = 3125$ is due to the isorropesis when the carbon atoms 5 and 10 are undergoing a make and break of a meta-linking, so that the band at $1/\lambda = 3220$ is due to the isorropesis occurring when the atoms 5 and 10 are undergoing a make and break of an ortho-linking.

We have been encouraged by these results to examine the absorption of some other aromatic hydrocarbons, namely, acenaphthene, naphthylene, fluorene, anthracene, chrysene, phenanthrene, benzyl, and diphenyl. The absorption curves of the first two are shown in Fig. 3 (full and dotted curves), and, as can be seen, are identical for the smaller concentrations, whilst at higher concentrations acenaphthylene shows a new set of bands (to which a visible colour is due). These last-mentioned bands must clearly be due to the isorropesis arising from the external double linking.

The very striking similarity between both these compounds and naphthalene is to be seen, for they exhibit very strongly the band at $1/\lambda = 3123$. This is the band which appears in α -tetrahydronaphthalene, so that the pulsating motions of acenaphthene and acenaphthylene must be very similar to those of naphthalene. This, however, is only to be expected from their structure, since they must have the two extreme phases respectively:

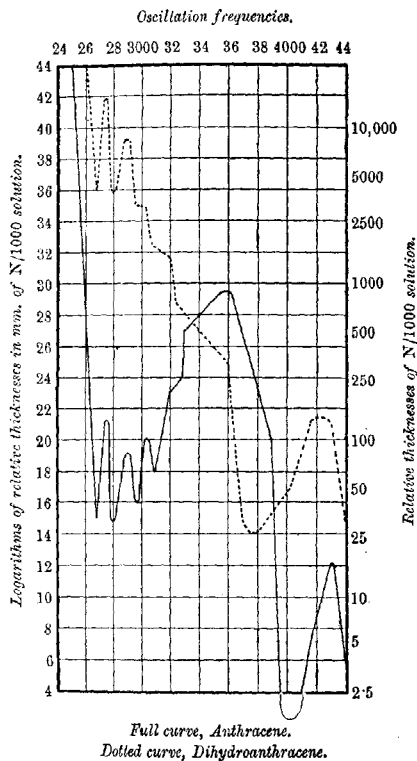


The curves of fluorene and anthracene are shown in Figs. 4 and 5 (all curves), and, as can be seen, show a marked similarity in that they both possess the four isorropesis bands at $1/\lambda = 2680, 2800, 3000$, and 3090 , although the shape of the curves are materially different in other respects. In Fig. 5 (dotted curve) is shown the

absorption curve of dihydroanthracene, which only differs from fluorene by the additional CH_2 group:

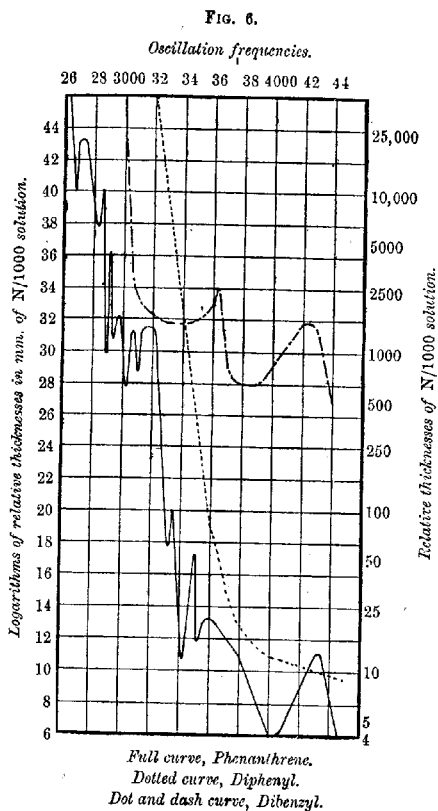


FIG. 5.



The curves of the two are very similar, but two of the isorropes of fluorene are almost absent in the curve of dihydroanthracene. The play of forces occurring in the three last compounds is dependent on the CH_2 group, which possesses a peculiar type of residual affinity which we propose to submit to future

investigation. This residual affinity depends on the substitution of the hydrogen atoms of methane by electro-negative groups, and evidences of this have already been observed in the case of benzyl alcohol and benzyl ethyl ether (Baly and Collie, *loc. cit.*), which both show small bands due to isorropesis between the phenyl group



and the side-chain. This effect is most strikingly exhibited in benzyl, the absorption curve of which is shown in Fig. 6 (dot and dash curve).

The absorption curves of chrysene and phenanthrene are shown in Fig. 3 (dot and dash curve) and Fig. 6 (full curve), and, although

certain regularities can be observed, the motions are too complex to permit of their elucidation until after the reduced derivatives have been examined.

In order to complete the series, we have included the absorption curve of diphenyl (Fig. 6, dotted curve), which only shows general absorption.

The absorption spectra of phenanthrene and anthracene have also been examined by Hartley (Trans., 1881, **39**, 153), and only minor differences are to be noted in our curves.

Conclusions.

1. The absorption spectrum of naphthalene shows three absorption bands, two narrow ones at $1/\lambda = 3125$ and 3220 , and a broad one at about $1/\lambda = 3700$. The third of these is due to the benzenoid motions of one ring, whilst the first two are due to the isorropesis between the benzenoid motions of one ring and the ethylenic system of double linkings of the other.

2. The isorropesis can be explained by adopting the view that the naphthalene molecule is in a state of pulsation like the benzene molecule. Evidence of this pulsation is especially strong in the cases of α -tetrahydronaphthalene, acenaphthene, and acenaphthylene.

3. The absorption spectra show that very similar play of forces are present in fluorene, anthracene, and dihydroanthracene. These are due to the presence of the CH_2 group, the inherent character of which is at present being studied.

Our thanks are due to Professor Collie for the interest he has taken in the investigation, and also to the Chemical Society for a grant in aid of the work.

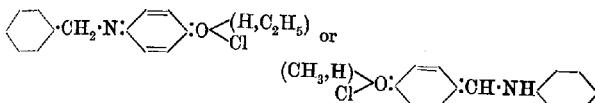
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CXCI.—*Colour and Constitution of Azomethine Compounds. Part II.*

By FRANK GEO. POPE and ROBERT FLEMING.

IN a previous communication (Trans., 1908, **93**, 532), it was shown by one of us that azomethine compounds containing a nitro- and hydroxyl group in the para-position to one another show an entire

different absorption spectrum from that exhibited by their sodium salts, and the conclusion was drawn that the alkali salts possess a quinonoid structure, whilst the free azomethine compounds contain the unaltered hydroxyl group. In the present investigation, the ethers of the azomethine series and the salts formed by the azomethines and their ethers with hydrochloric acid have been examined. The azomethines and their ethers show similar absorptions in neutral solvents, whilst the hydrochlorides, which are almost without exception deeply coloured (bright yellow) compounds, show absorption in quite a different region of the spectrum. The simplest members of the series are colourless, crystalline solids, and show no absorption in the visible spectrum, whilst the hydrochlorides, on the other hand, shows a distinct band in the blue and violet regions of the spectrum. Thus it seems necessary to conclude that the salts of the azomethines and their ethers must possess a different structure from the parent substances, and in the opinion of the authors it seems most rational to regard them as oxonium salts, thus:



This formula also brings the azomethine salts into agreement with Hewitt and Mitchell's observation (Trans., 1907, 91, 1251), that the lengthening of the chain of alternate double and single linkings accompanies the shifting of the absorption to the red end of the spectrum.

The hydrochlorides described were prepared by dissolving the azomethines in pure benzene and saturating the solution with hydrogen chloride. The precipitated hydrochloride was collected, washed with light petroleum or benzene, and dried on a porous tile. In most cases the hydrochlorides obtained were stable, but in one or two instances analytical data have not been obtained, owing to the instability of the compounds.

EXPERIMENTAL

Benzylidene-p-aminophenol hydrochloride, $\text{C}_6\text{H}_5 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{OH} \cdot \text{HCl}$, is obtained as a yellow, microcrystalline powder, which decomposes when heated to 132° . It is readily soluble in hot alcohol:

0.3562 gave 0.2142 AgCl. $\text{HCl} = 15.34$.

$\text{C}_{13}\text{H}_{11}\text{ON} \cdot \text{HCl}$ requires $\text{HCl} = 15.63$ per cent.

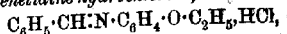
Benzylidene-p-amisidine hydrochloride, $\text{C}_6\text{H}_5 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{CH}_3 \cdot \text{HCl}$, is a sulphur-yellow, microcrystalline powder. It melts at 70° :

0.3412 gave 0.1886 AgCl. $\text{HCl} = 14.06$.

$\text{C}_{14}\text{H}_{13}\text{ON} \cdot \text{HCl}$ requires $\text{HCl} = 14.55$ per cent.

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Benzylidene-p-phenetidine hydrochloride,

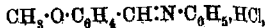


is a pale yellow, crystalline powder. It decomposes at 138—139°:

0.3455 gave 0.1888 AgCl. $\text{HCl} = 13.90$.

$\text{C}_{15}\text{H}_{16}\text{ON}, \text{HCl}$ requires $\text{HCl} = 13.96$ per cent.

Anisylideneaniline hydrochloride,



crystallises in minute, sulphur-yellow needles, which decompose at 114°:

0.175 gave 0.1016 AgCl. $\text{HCl} = 14.75$.

$\text{C}_{14}\text{H}_{13}\text{ON}, \text{HCl}$ requires $\text{HCl} = 14.55$ per cent.

Anisylidene- α -naphthylamine, $\text{CH}_3 \cdot \text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{N} \cdot \text{C}_{10}\text{H}_7$, is obtained when equivalent quantities of anisaldehyde and α -naphthylamine are dissolved in absolute alcohol and the solutions mixed together. It is precipitated by the addition of water, collected, washed with water, and finally recrystallised from hot alcohol. It forms small, colourless, glistening plates, which melt at 100—101°:

0.2432 gave 0.7374 CO_2 and 0.1292 H_2O . $\text{C} = 82.68$; $\text{H} = 5.90$.

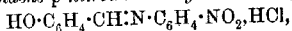
$\text{C}_{15}\text{H}_{15}\text{ON}$ requires $\text{C} = 82.76$; $\text{H} = 5.75$ per cent.

The hydrochloride separates as a dark yellow, semi-crystalline powder, which gradually decomposes on exposure. It decomposes when heated to 211°:

0.3386 gave 0.1482 AgCl. $\text{HCl} = 11.21$.

$\text{C}_{15}\text{H}_{15}\text{ON}, \text{HCl}$ requires $\text{HCl} = 12.2$ per cent.

o-Hydroxybenzylidene-p-nitroaniline hydrochloride,

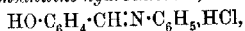


separates as an orange-yellow powder, which melts and decomposes at 179—180°:

0.2851 gave 0.1435 AgCl. $\text{HCl} = 12.8$.

$\text{C}_{13}\text{H}_{10}\text{O}_2\text{N}_2, \text{HCl}$ requires $\text{HCl} = 13.10$ per cent.

o-Hydroxybenzylideneaniline hydrochloride,



forms pale yellow, microscopic needles, which melt at 91°:

0.6198 gave 0.3774 AgCl. $\text{HCl} = 15.47$.

$\text{C}_{13}\text{H}_{11}\text{ON}, \text{HCl}$ requires $\text{HCl} = 15.63$ per cent.

o-Hydroxybenzylidene- α -naphthylamine, $\text{HO} \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{N} \cdot \text{C}_{10}\text{H}_7$, is obtained when equivalent quantities of salicylaldehyde and α -naphthylamine are separately dissolved in alcohol, the solutions mixed, and allowed to stand for some time. On the addition of water to the solution, an oil separates. On pouring off the supernatant liquor and allowing the oil to stand for some time, it gradually crystallises in stellate groups of needles. These were dried and recrystallised from

enzene, from which they separate in pale yellow needles, melting at 53° :

0.3628 gave 1.0972 CO_2 and 0.1724 H_2O . $\text{C} = 82.48$; $\text{H} = 5.27$.

$\text{C}_{17}\text{H}_{13}\text{ON}$ requires $\text{C} = 82.59$; $\text{H} = 5.27$ per cent.

The *hydrochloride*, on precipitation from a benzene solution, separates first as a semi-liquid mass, but, on standing for some time, gradually solidifies to a bright yellow, amorphous powder, which decomposes at 110° :

0.5235 gave 0.2724 AgCl . $\text{HCl} = 13.19$.

$\text{C}_{17}\text{H}_{13}\text{ON} \cdot \text{HCl}$ requires $\text{HCl} = 12.88$ per cent.

p-Nitrobenzylidene-o-anisidine, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{CH}_3$, is obtained by warming equivalent quantities of its generators in alcoholic solution. On cooling, a yellow, crystalline mass separates. This is collected, washed with very dilute alcohol, and finally recrystallised from dilute alcohol. It forms sulphur-yellow plates, which melt at 111° :

0.2386 gave 0.5484 CO_2 and 0.0999 H_2O . $\text{C} = 65.43$; $\text{H} = 4.42$.

$\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}_2$ requires $\text{C} = 65.62$; $\text{H} = 4.68$ per cent.

The *hydrochloride* is a very pale yellow, amorphous powder, which decomposes at 141° :

0.1874 gave 0.086 AgCl . $\text{HCl} = 11.67$.

$\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}_2 \cdot \text{HCl}$ requires $\text{HCl} = 12.47$ per cent.

p-Nitrobenzylidene-p-anisidine, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{CH}_3$, is obtained by mixing equivalent quantities of its generators in alcoholic solution. The mixture sets to a mass of yellow crystals. These are collected, well washed, and recrystallised from absolute alcohol. The compound is thus obtained in golden-yellow leaflets, which melt at 39° , and are only sparingly soluble in hot alcohol :

0.1944 gave 0.466 CO_2 and 0.0856 H_2O . $\text{C} = 65.38$; $\text{H} = 4.9$.

$\text{C}_{14}\text{H}_{12}\text{O}_3\text{N}_2$ requires $\text{C} = 65.62$; $\text{H} = 4.68$ per cent.

The *hydrochloride* forms a mass of microscopic, sulphur-yellow crystals, which decompose when heated to 200° :

0.1886 gave 0.0975 AgCl . $\text{HCl} = 13.14$.

$\text{C}_{14}\text{H}_{12}\text{O}_3\text{N}_2 \cdot \text{HCl}$ requires $\text{HCl} = 12.47$ per cent.

p-Nitrobenzylidene-p-phenetidine, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{C}_2\text{H}_5$, is prepared by a similar method to the above anisidine compound. It crystallises in sulphur-yellow needles, which melt at 130.5° , and are only sparingly soluble in hot alcohol :

0.1701 gave 0.4174 CO_2 and 0.0836 H_2O . $\text{C} = 66.92$; $\text{H} = 5.50$.

$\text{C}_{15}\text{H}_{14}\text{O}_3\text{N}_2$ requires $\text{C} = 66.67$; $\text{H} = 5.18$ per cent.

The *hydrochloride* is obtained in small, bright yellow needles, which decompose at 196° :

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0.2354 gave 0.105 AgCl. $\text{HCl} = 11.34$.
 $\text{C}_{15}\text{H}_{14}\text{O}_3\text{N}_2$, HCl requires $\text{HCl} = 11.90$ per cent. *

p-Nitrobenzylidene-1-amino- β -naphthol hydrochloride,
 $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_{10}\text{H}_6 \cdot \text{OH} \cdot \text{HCl}$,
 forms small, brownish-coloured needles, which decompose at $230-235^\circ$.

0.1534 gave 0.0688 AgCl. $\text{HCl} = 11.40$.
 $\text{C}_{17}\text{H}_{12}\text{O}_3\text{N}_2$, HCl requires $\text{HCl} = 11.11$ per cent.

o-Nitrobenzylidene-*p*-aminophenol, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{OH}$, is obtained by mixing equivalent quantities of its generators in dilute acetic acid solution. On the addition of water to the solution, a pale yellow precipitate is formed. This is collected, washed, and crystallised from dilute alcohol, from which it separates in long, yellow needles melting at 159° :

0.2392 gave 0.5595 CO_2 and 0.0902 H_2O . $\text{C} = 63.79$; $\text{H} = 4.22$.
 $\text{C}_{13}\text{H}_{10}\text{O}_3\text{N}_2$ requires $\text{C} = 64.46$; $\text{H} = 4.13$ per cent.

The hydrochloride is a bright yellow, amorphous powder, which decomposes at 216° :

0.2758 gave 0.1398 AgCl. $\text{HCl} = 12.89$.
 $\text{C}_{13}\text{H}_{10}\text{O}_3\text{N}_2$, HCl requires $\text{HCl} = 13.11$ per cent.

2 : 4-Dinitrobenzylidene-*p*-phenetidine,
 $(\text{NO}_2)_2\text{C}_6\text{H}_3 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{O} \cdot \text{C}_2\text{H}_5$,

is prepared by mixing equivalent quantities of its generators in hot alcohol. On the addition of water, a brownish-yellow precipitate is obtained. This is collected, well washed, and crystallised from much hot alcohol. It forms tufts of brownish-yellow needles which melt at 135° and are only very sparingly soluble in hot alcohol or benzene. It undergoes a certain amount of decomposition on exposure:

0.137 gave 0.2848 CO_2 and 0.0554 H_2O . $\text{C} = 56.69$; $\text{H} = 4.52$.
 $\text{C}_{15}\text{H}_{13}\text{O}_5\text{N}_2$ requires $\text{C} = 57.14$; $\text{H} = 4.12$ per cent.

p-Nitrobenzylidene-2-nitro-*p*-phenetidine,
 $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} : \text{N} \cdot \text{C}_6\text{H}_3(\text{NO}_2) \cdot \text{O} \cdot \text{C}_2\text{H}_5$,

is obtained by mixing its generators in hot alcohol. It separates on the addition of water as a mass of cinnabar-red crystals. These are collected, well washed, and crystallised from dilute alcohol, from which deep orange-red needles melting at 84° are obtained:

0.1445 gave 0.301 CO_2 and 0.0583 H_2O . $\text{C} = 56.75$; $\text{H} = 4.48$.
 $\text{C}_{15}\text{H}_{13}\text{O}_5\text{N}_2$ requires $\text{C} = 57.14$; $\text{H} = 4.13$ per cent.

On passing hydrogen chloride into a benzene solution of the above compound, a greyish-coloured precipitate is formed, which is in all probability the hydrochloride, but no analytical data could be obtained.

since on exposure to air the substance regenerates the deep red azomethine compound with great rapidity.

The *hydrochloride* of *p*-nitrobenzylidene-*p*-aminophenol has also been prepared, but not as yet in a pure condition. It is a buff-coloured powder, but apparently undergoes a certain amount of decomposition when drying, since, on analysis, it gave the following numbers:

0.4334 gave 0.2479 AgCl. HCl = 14.54

0.3638 „ 0.2098 AgCl. HCl = 14.67.

$C_{18}H_{10}O_2N_2$, HCl requires HCl = 13.11 per cent.

Our thanks are due to Mr. O. R. Howells for much assistance with the analytical data, and to the Research Fund Committee of the Chemical Society for a grant which has partly defrayed the expense of this work.

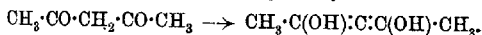
EAST LONDON COLLEGE.

CXCII.—*The Relation between Viscosity and Chemical Constitution. Part III. The Enol-ketonic Tautomerism.*

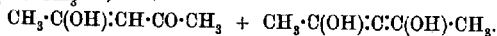
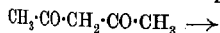
By ALBERT ERNEST DUNSTAN and JAMES ARTHUR STUBBS.

MOST of the evidence derived from the physical examination of automeric compounds of the ethyl acetoacetate type shows that in the ordinary liquid condition the ketonic form predominates.

Brühl (*Zeitsch. physikal. Chem.*, 1900, **34**, 31), from determinations of the molecular refractivity and molecular dispersion, concluded that the free ester is mainly ketonic, whereas its sodium compound is enolic. Moreover, he showed that negative groups favour the enolic structure; thus acetylacetone is most probably dienolic:



Perkin (*Trans.*, 1892, **61**, 800) also concludes that ethyl acetoacetate is a true ketone, and that acetylacetone consists of 30 per cent. of the mono- and 70 per cent. of the dienolic forms:



The same observer (*loc. cit.*) points out that ethyl acetylacetone is ketonic, $CH_3 \cdot CO \cdot CH(C_2H_5) \cdot CO \cdot CH_3$, and maintains that positive radicals favour the ketonic structure. The ketonic modification is

also formed in increasing amounts when the temperature rises, pointing to a state of equilibrium which is influenced by temperature.

Schaum (*Ber.*, 1898, 31, 1964) determined the molecular solution volume in many solvents, and showed that the gradual change in this property was due to a change in the equilibrium.

Drude (*Ber.*, 1897, 30, 940), in his observations on anomalous electric absorption, demonstrated that ethyl acetoacetate was in the main ketonic, since the high frequency radiation was not absorbed, as would be the case for an hydroxylated compound.

In general, it has been found that the change from the ketonic to the enolic form is accelerated by bases, and the reverse change brought about by such solvents as chloroform; thus Knorr (*Annalen*, 1896, 293, 70) converted the enolic dibenzoylsuccinic ester into the ketonic form by simple heating, and formylphenylacetic ester from the enolic to the ketonic form by the action of water, but effected the reverse change by means of benzene.

Lowry (*Trans.*, 1899, 75, 241) explains the ketonising effect of piperidine, and the enolising effect of sodium ethoxide, by assuming a loose combination between the reagent and the enol-ketonic compound. Each of these loose compounds may then react with a third substance present, for example, benzylideneaniline, liberating the piperidine or sodium ethoxide, which is then available for further transformation.

Our own results support Lowry's view so far as piperidine is concerned, for the marked increase in viscosity when piperidine is added in increasing amounts to ethyl acetoacetate is evidently due to combination between the two substances.

In the present communication, we have determined the viscosity of ethyl acetoacetate alone, and in solution with benzene, pyridine, alcohol, and 50 per cent. aqueous alcohol as solvents, also of ethyl ethylacetoacetate and ethyl diethylacetoacetate alone, and in solution with benzene, alcohol, and pyridine as solvents.

We have also investigated the effects of time on the viscosity of acetoacetic ester, and the result of adding piperidine and sodium ethoxide to the pure ester. A summary of the investigation will be found at the end of the paper.

The pure materials used were supplied by Kahlbaum, and were purchased with a grant from the Research Committee of the Chemical Society, to whom we make this grateful acknowledgment.

EXPERIMENTAL.

The details of the methods employed will be found in our previous communications. All determinations were carried out at 25°; the densities corrected for displaced air are given with respect to water at 4°. The solvents were carefully purified by distillation over appropriate dehydrating agents.

Ethyl Acetoacetate (b. p. 80°/20 mm.) and *Benzene* (b. p. 80.4°).

Per cent. ester.	Time of flow, in seconds.	Density 25°/4°.	Viscosity.
0.0	42.2	0.8736	0.006145
2.78	42.3	0.8770	0.006170
10.47	43.4	0.8869	0.006147
43.56	51.1	0.8824	0.007942
98.83	83.4	1.0126	0.01407
100.0	88.5	1.0222	0.015081

The curve falls into class I of our system (Trans., 1907, 91, 83), and indicates that little mutual action has proceeded in the solution.

Ethyl Acetoacetate and Ethyl Alcohol (b. p. 77.8°/755 mm.).

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.0	81.3	0.7875	0.01967
8.05	76.5	0.8025	0.01023
36.28	67.1	0.8605	0.009625
46.71	66.5	0.8832	0.009791
64.02	67.3	0.9244	0.010370
100.0	88.5	1.0222	0.015081

This curve belongs to class 3, and exhibits a minimum point of precisely similar nature to those given by alcohol and benzene (Trans., 1904, 85, 822). It should be noticed particularly that mixtures of acetone and alcohol afford a type of curve which very closely resembles that of ethyl diethylacetoacetate and alcohol, and has only a general resemblance to that furnished by ethyl acetoacetate and alcohol. The full significance of this will appear in the sequel.

Ethyl Acetoacetate and Pyridine (b. p. 115°).

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	54.0	0.97817	0.008805
17.11	56.8	0.9853	0.009297
32.09	60.0	0.9916	0.009918
51.00	65.4	0.9999	0.01090
71.07	73.3	1.0091	0.01233
100.00	88.5	1.0222	0.015081

This curve belongs to class 1.

Ethyl Acetoacetate and 2:6-Lutidine (b. p. 173°).

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	54.6	0.9324	0.008885
10.9	56.6	0.9413	0.009298
32.33	60.8	0.9598	0.01018
52.01	67.2	0.9769	0.01146
88.55	81.6	1.0112	0.01439
100.0	86.1	1.0222	0.01536

This curve belongs to class 1.

Ethyl Acetoacetate and 50 per cent. Aqueous Alcohol.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	145.0	0.9101	0.02303
14.13	131.6	0.9243	0.02123
32.32	119.2	0.9444	0.01965
61.23	102.8	0.9770	0.01753
100.00	86.1	1.0222	0.01536

This curve belongs to class 1.

Ethyl Ethylacetoacetate (b. p. 198°) and Benzene.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	42.2	0.8736	0.006145
17.75	45.2	0.8907	0.006711
24.98	47.3	0.8974	0.007076
38.13	51.0	0.9086	0.007725
100.00	102.5	0.9754	0.01667

This curve is of the same type as that given by the ethyl acetoacetate, but the sagging is less pronounced.

Ethyl Ethylacetoacetate and Ethyl Alcohol.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	78.6	0.7865	0.01079
5.44	76.7	0.7953	0.01064
23.80	71.9	0.8271	0.01038
41.24	70.6	0.8588	0.01058
71.31	75.2	0.9165	0.01203
87.87	83.6	0.9495	0.01385
100.00	98.7	0.9750	0.01679

This curve belongs to class 3, as does that of the ethylacetoacetate, but the minimum is less pronounced.

Ethyl Ethylacetoacetate and Pyridine.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	51.9	0.9742	0.008523
12.70	55.0	0.9743	0.009352
40.62	64.7	0.9750	0.01101
63.99	74.5	0.9755	0.01268
85.33	87.6	0.9751	0.01491
100.0	98.7	0.9750	0.01679

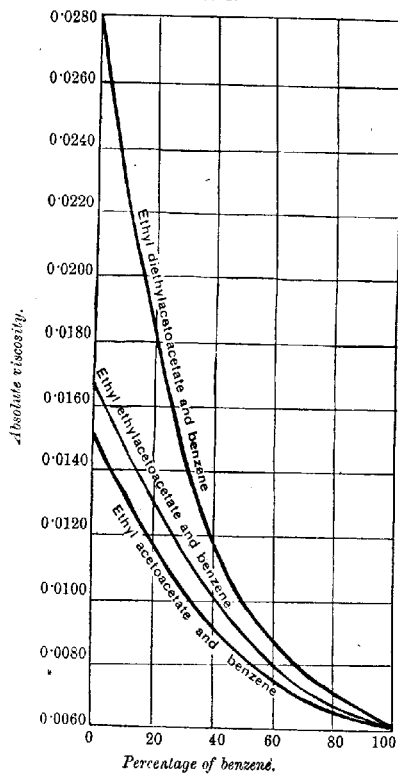
This curve belongs to class 1, and closely resembles that of the ethyl acetoacetate.

Ethyl Diethylacetoacetate (b. p. 218°) and *Alcohol*.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	81.3	0.7875	0.01067
15.66	80.9	0.8112	0.01094
20.74	81.0	0.8200	0.01107
28.56	82.0	0.8325	0.01138
91.78	138.3	0.9491	0.02188
100.0	178.7	0.9646	0.02793

This curve is of class 1, but has a similar sag to that of acetone and alcohol.

FIG. 1.

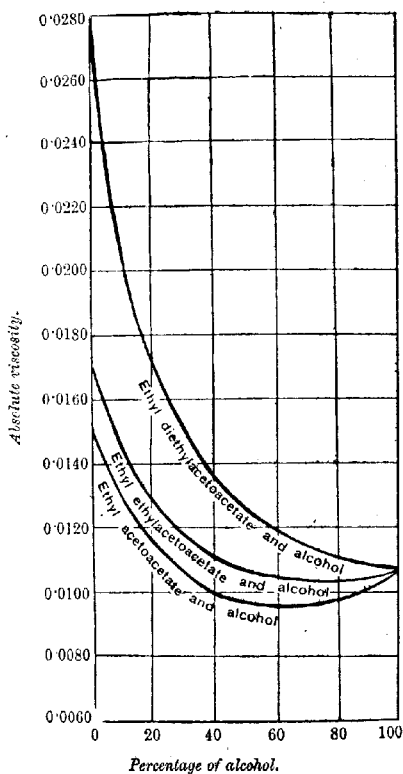


Ethyl Diethylacetoacetate and Benzene.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	42.2	0.8736	0.006145
3.65	42.7	0.8766	0.006240
14.16	46.2	0.8851	0.006817
49.52	64.6	0.9171	0.009876
68.19	84.8	0.9345	0.01321
100.00	173.7	0.9646	0.02793

This curve is in class 1.

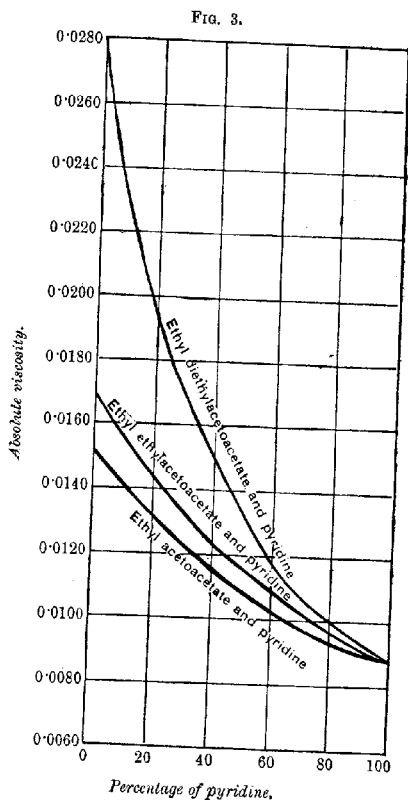
FIG. 2.



Ethyl Diethylacetoacetate and Pyridine.

Per cent. ester.	Time of flow.	Density 25°/4°.	Viscosity.
0.00	54.0	0.9782	0.008805
2.83	55.0	0.9781	0.008967
6.91	56.6	0.9777	0.009225
21.10	62.4	0.9758	0.01015
50.83	83.2	0.9719	0.01348
100.00	173.7	0.9646	0.02793

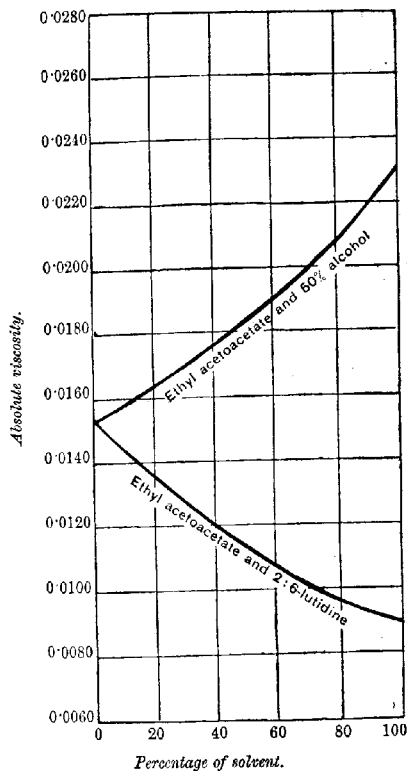
This curve is in class 1.



The effect of piperidine on ethyl acetoacetate was next studied. The pure ester had a time of flow of 87.2 seconds. On adding 4.5 per

cent. piperidine, the time of flow was 98.6 seconds, and when the concentration reached 7.5 per cent., the time of flow became 107.4 seconds. For piperidine itself, the time of flow was 124.2 seconds. This gives a curve which would fall into class 2, and is indicative of a combination between piperidine and the ester.

FIG. 4.



In a similar way, a trace of sodium ethoxide raised the time of flow from 87.2 to 87.8 seconds. The ester (time of flow, 87.2 seconds), on redistillation, gave a time of flow of 87.7 seconds. This is in accord with the observation of Schaum (see introduction).

Comparison of the Curves (Figs. 1, 2, 3, 4).

In Fig. 1 are plotted together the results of the measurements in benzene solution. It is obvious that a close similarity obtains, a fact that points to a similarity of structure in the three esters. Mention should be made here of the striking difference in viscosity between the diethyl ester and the other two. Now, if ethyl acetoacetate were as completely ketonic as the ethyl derivatives, we should expect a considerable viscosity interval between ethyl acetoacetate and ethyl ethylacetoacetate. The viscosity curve of the former, however, approximates to the latter, which is explicable according to the views we have brought forward from time to time on the assumption that ethyl acetoacetate is partly enolic.

Fig. 2 illustrates the results obtained from the alcoholic solutions. Now, one of us showed (Trans., 1904, 85, 820) that the acetone-alcohol mixture is an exaggerated variety of class 1, that is, the sag is most prominent and verges on a minimum point.

The three curves in this diagram bear out the resemblance to the acetone curve, greater in the case of the diethyl ester, less in the case of ethyl acetoacetate itself. Again, the same conclusion may be drawn. The diethyl ester is the most ketonic, ethyl acetoacetate being partly (and considerably) ketonic.

Fig. 3 contains the data afforded by the pyridine solutions. The same point is brought out here again; the three curves are of the same type; apparently pyridine ketonises the ethyl acetoacetate as factually as does benzene.

Fig. 4 demonstrates that both in 50 per cent. alcohol and in 6:4 lutidine the ketonic form predominates.

We are extending this investigation to other instances of tautomeric substances.

PHYSICAL CHEMISTRY LABORATORY,
EAST HAM TECHNICAL COLLEGE.

CXCIII.—*Contributions to the Chemistry of the Cholesterol Group. Part II. Some Oxidation Products of Sitosterol.*

By ROBERT HOWSON PICKARD and JOSEPH YATES.

By the action of hydrogen peroxide on cholesterol (see Part I, this vol., 1678), it is possible to obtain an almost quantitative yield of the first product of the oxidation of this alcohol. This splendid reaction induced us to investigate the applicability of the reagent to a member of this group of alcohols derived from the vegetable kingdom. These compounds are found only in small amounts, but are very widely distributed in plant life. Perhaps the best source is the so-called wheat "germs," once a waste product of the flour-mill, but now used as a constituent of various patent breads. The fat from wheat "germs," as shown by Burian (*Monatsh.*, 1897, **18**, 551), contains an alcohol named by him *sitosterol*, which is closely allied to cholesterol. Researches by Windaus and Hanth (*Ber.*, 1907, **40**, 3681) on the fat derived from Calabar beans seem to indicate that sitosterol is a constituent of most vegetable cholesterol (the phytosterols).

Sitosterol behaves with hydrogen peroxide in a very similar manner to cholesterol, and easily yields a triol.

Having prepared by this reaction a quantity of the initial product of the oxidation of sitosterol, we proceeded to prepare some further oxidation products by methods partly analogous to those applied to the cholesteryl derivatives by other investigators (see Part I, *loc. cit.*; Manthner and Suida, *Monatsh.*, 1896, **17**, 579, and Windaus, *Ber.*, 1906, **36**, 2754). We have found that there is a great similarity in the behaviour of the two series of compounds, but no members of the two series are identical. This analogous behaviour of the sitosteryl derivatives suggests that this alcohol, like cholesterol (compare Windaus, *loc. cit.*), possesses a terminal vinyl group.

The compounds obtained are tabulated below, with the melting points and empirical formulæ compared with those of the corresponding cholesteryl derivatives, but it must be remembered that the analysis of all these compounds is difficult (see Part I), and the results somewhat ambiguous.

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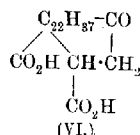
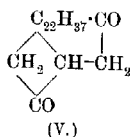
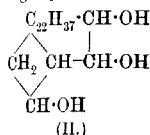
Name.*	M. p.	Formula,	M. p. of corresponding cholesteryl derivative.
I. Sitosterol	138 ²	C ₂₇ H ₄₆ O	147 ²
" acetate	128		113
" phenylcarbamate	158		168
II. Dehydrositostanetriol	252	C ₂₇ H ₄₆ O ₃	239 ¹
" diacetate.....	138—139		165 ¹
III. Dehydrositostanedione	256	C ₂₇ H ₄₂ O ₃	231 ²
IV. Dehydrositostanedione	166	C ₂₇ H ₄₀ O ₂	122 ²
Monophenylhydrazine	247		271 ²
V. Dehydrositostanedione	196	C ₂₇ H ₄₂ O ₂	169 ³
" dioxime.....	187		205 ³
VI. Ketodicarboxylic acid.....	236	C ₂₇ H ₄₂ O ₃	219 ²
" dimethyl ester.....	107		113—114 ²
Oxime of ester	90 (about)		76 ³

¹ Part I.

² Mauthner and Suida, *loc. cit.*

³ Windaus, *loc. cit.*

The relationship between some of the cholesteryl derivatives is formulated in Part I (*loc. cit.*). A similar relationship is probable in the case of the sitosteryl derivatives, that between II, V, and VI being represented below :



EXPERIMENTAL.

Source of Sitosterol.

The sitosterol used in these experiments was prepared from wheat germs (compare Burian, *loc. cit.*). These were extracted first with oiling methylated spirit, and, after removal of the solvent, the viscous mass was agitated with ether to separate the fat from other substances extracted by the alcohol.

The fat thus obtained was hydrolysed by potassium hydroxide in alcoholic solution until a test portion dissolved completely in water. The alcohol was then distilled off, and the product dissolved in hot water. Calcium chloride solution was then added until no further precipitation occurred, when the insoluble calcium salt and unsaponifiable matter were removed by filtration and thoroughly dried. This product was then extracted four or five times with boiling acetone. Owing to the presence of undecomposed fat in the product left after removal of the acetone, the hydrolysis and subsequent treatment were repeated two or three times.

* A nomenclature analogous to that by Windaus (*Arch. Pharm.*, 1903, 246, 117) for cholesteryl derivatives is followed throughout.

The sitosterol ultimately obtained was crystallised from alcohol, dried, and converted into the acetate by boiling for thirty minutes with its own weight of acetic anhydride. The sitosterol acetate (m. p. 128°) was then hydrolysed, and the resulting sitosterol crystallised several times from alcohol. It crystallises in lustrous plates, which are similar in appearance to cholesterol and melt at 138° . After heating at 100° , the substance had $[\alpha]_D - 34.4^{\circ}$ (with $c=5.5$ in chloroform) and $[\alpha]_D - 23.21^{\circ}$ (with $c=2.9$ in ether).

The amount obtained from two sacks of "germ" (representing about 200 tons of wheat) was 60 grams.

Sitosteryl Phenylcarbamate.—The phenylcarbamates of these alcohols are probably the derivatives best suited for their characterisation, since they are quickly prepared and are easier to crystallise than the benzoates which have hitherto been employed. *Sitosteryl phenylcarbamate* was prepared by heating together equivalent quantities of sitosterol and phenylcarbimide for two hours at 160° . It crystallises readily from glacial acetic acid in very lustrous plates or needles, which melt at 158° :

0.2470 gave 6.2 c.c. N_2 (moist) at 21° and 734 mm. $N=2.74$.

$C_{84}H_{149}O_3N$ requires $N=2.78$ per cent.

0.9745, made up to 25 c.c. with benzene, gave $\alpha - 2.03^{\circ}$; whence $[\alpha]_D^{25} - 26.03^{\circ}$.

Dehydrositostanetriol.

A mixture of sitosteryl acetate (36 grams), glacial acetic acid (250 c.c.), and hydrogen peroxide (20 c.c. of Merck's perhydrol) was heated on the water-bath for one hour. An oil separated out at first, but, after a short time, the mixture became clear, and when cold deposited a solid, which, when dry, melted indefinitely at about 210° . Water was added to the filtrate, and precipitated a pasty solid, which, with the other product, was heated for three hours with an excess of an alcoholic solution of sodium ethoxide. The mixture was then poured into water, and the resulting precipitate, when dry, extracted three times with hot benzene. The insoluble residue left was the pure triol melting sharply at 252° . When cold, the benzene solution deposited a further quantity of the triol, the total yield of which amounted to about 60 per cent., whilst unaltered sitosterol was recovered from the benzene filtrate.

The triol is more or less soluble in the common organic media with the exception of light petroleum. It is very sparingly soluble in cold benzene, from which it crystallises in masses of felted needles which melt at 252° :

0.1196 gave 0.3370 CO_2 and 0.1227 H_2O . $C=76.9$; $H=11.4$.

$C_{27}H_{46}O_3$ requires $C=77.5$; $H=11.0$ per cent.

Diacetate.—The diacetate is easily prepared by heating the triol with acetic anhydride and sodium acetate for thirty minutes. It is soluble in most organic media, except light petroleum. The best crystallising medium is acetic anhydride, from which it separates in clusters of needles melting at 138—139°.

0.6501, boiled with a solution of sodium hydroxide, neutralised 0.0992 NaOH. The calculated amount for $C_{27}H_{44}O_5Ac_2$ is 0.1036.

The *propionyl* and *benzoyl* derivatives were prepared, but crystallised badly. All the acyl derivatives yielded the triol when hydrolysed.

Dehydrositostanedionol.

Eight grams of the triol were dissolved in 600 c.c. of glacial acetic acid and the solution cooled to 40°, when a solution of 8 grams of chromium trioxide in 100 c.c. of 90 per cent. acetic acid were added. After twenty-four hours, about 5 grams of a crystalline precipitate were obtained. The dionol crystallises from acetic acid in masses of tilted needles, which melt at 256° and are only sparingly soluble in the common organic media:

0.1227 gave 0.3510 CO_2 and 0.1192 H_2O . $C = 78.02$; $H = 10.7$.

$C_{27}H_{42}O_3$ requires $C = 78.29$; $H = 10.1$ per cent.

Dehydrositostenedione.

The dionol readily loses the elements of water, and yields an unsaturated ketone. The reaction is carried out as follows: The dionol suspended in chloroform is treated with dry hydrogen chloride in the cold, when it slowly dissolves, forming a yellow solution. After thirty minutes, the solution is washed with water and dried. On removal of the solvent, the unsaturated ketone remains as a yellow, crystalline mass. It is soluble in most solvents, and crystallises well from methyl alcohol in yellow leaflets, which melt at 166°:

0.1202 gave 0.3616 CO_2 and 0.1143 H_2O . $C = 82.0$; $H = 10.5$.

$C_{27}H_{40}O_2$ requires $C = 81.8$; $H = 10.1$ per cent.

The substance after some time becomes much lighter in colour, eventually a very pale yellow. It gives a red coloration with glacial acetic acid, and separates from this solvent in pale red crystals, which gradually lose their colour on standing. The melting points of all these differently tinted varieties were the same, and none of them gave a coloration with an ethereal solution of ferric chloride. It is optically active, and had in chloroform solution $[\alpha]_D - 38.9^\circ$.

Monophenylhydrazone.—An alcoholic solution warmed with a few

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drops of phenylhydrazine deposits a yellow *phenylhydrazane*, which crystallises readily from acetic acid in transparent, pale yellow leaflets melting at 247° :

0.1274 gave 6.8 c.c. N_2 (moist) at 18° and 736 mm. $N = 5.9$,
 $C_{33}H_{46}ON_2$ requires $N = 5.7$ per cent.

Dehydrositostanedione.

A solution of the above unsaturated ketone in acetic acid was heated with zinc dust for four hours, when the solution had lost its red colour. On pouring into water, a white precipitate was obtained which crystallised from ethyl alcohol in hard nodules melting at 196° :

0.1166 gave 0.3449 CO_2 and 0.1173 H_2O . $C = 80.7$; $H = 11.1$,
 $C_{27}H_{43}O_2$ requires $C = 81.4$; $H = 10.6$ per cent.

Dioxime.—The diketone treated in the usual manner with an alcoholic solution of hydroxylamine gave the *dioxime*, which crystallises from benzene in slender, silky needles, or from methyl alcohol in stout prisms, and melts at 187° :

0.1154 gave 7.0 c.c. N_2 (moist) at 19° and 752 mm. $N = 6.8$,
 $C_{27}H_{44}O_2N_2$ requires $N = 6.5$ per cent.

Acid from Oxidation of Dehydrositostanedione.

When the dione is heated under a reflux condenser with an acetic acid solution of its own weight of chromium trioxide, it is converted into a dibasic ketonic acid with the probable empirical formula $C_{27}H_{43}O_5$. It crystallises from glacial acetic acid in transparent leaflets, which melt and decompose at 236° :

0.2490 neutralised 0.0408 NaOH. The calculated quantity for a dibasic acid, $C_{27}H_{43}O_5$, is 0.0446 :

0.1191 gave 0.3175 CO_2 and 0.1062 H_2O . $C = 72.7$; $H = 9.9$,
 $C_{27}H_{43}O_5$ requires $C = 72.6$; $H = 9.4$ per cent.

The acid is insoluble in water and light petroleum, and very sparingly soluble in benzene.

Dimethyl Ester.—The acid, when warmed with methyl alcohol and a few drops of concentrated sulphuric acid, yields a neutral ester, which crystallises from methyl alcohol in needles melting at 107° .

The ester forms an *oxime*, which melts at about 90° , but the quantity of material remaining did not allow of its analysis or further experiments with the substance.

It appears possible that the reactions of the cholesterol with hydrogen peroxide may prove of service in analytical operations where

the detection of animal and vegetable fats is required. The extreme insolubility of the triols in cold benzene affords a ready means for the detection of cholesterol in admixture with various fats. We have carried out several preliminary experiments with wool-fat and butter, which were very encouraging, but we do not propose to experiment further in the analytical direction.

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XLIV.—The Condensation of Salicylaldehyde and Benzamide.

By ARTHUR WALSH TITHERLEY and MORRIS EDGAR MARPLES.

THE condensation of salicylaldehyde with amides has been studied by Cebrian (*Ber.*, 1898, **31**, 1592), who described a series of cyclic derivatives, which he named "coumarazines," and to which he gave the

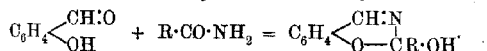
general formula $C_6H_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \text{O} \diagdown \\ \text{CR}\cdot\text{OH} \end{smallmatrix}$. Owing to the similarity in

structure of these coumarazines to the metoxazone derivatives which one of the authors has shown represent the tautomeric form of *O*- and

N-acylsalicylamides, $(C_6H_4 \begin{smallmatrix} \text{CO}\cdot\text{NH} \\ \diagup \text{O} \diagdown \\ \text{CR}\cdot\text{OH} \end{smallmatrix})$, where there is a similar

hydroxylated metoxazine skeleton present, it was thought desirable to submit Cebrian's derivatives to close study. Auwers (*Ber.*, 1907, **40**, 3510), in criticising the theory of metoxazone tautomerism as applied to the acylsalicylamides, drew attention to the difference between the crystalline character of the latter and the amorphous properties of the coumarazines, and considered that this difference precluded the possibility of the acylsalicylamides appearing in a metoxazone form.

Repetition of Cebrian's work, however, brought to light the fact that his so-called coumarazines are not really cyclic in structure, but that they are simply open-chain condensation derivatives, having a phenolic character similar to the condensation product (benzylidene-salicylamide) obtained by the action of benzaldehyde on salicylamide (Titherley, *Trans.*, 1907, **91**, 1420). Cebrian, who considered the possibility of an open-chain formula, $HO\cdot C_6H_4\cdot CH:N\cdot COR$, for his products, was led to adopt a ring formula according to the reaction:



His reasons were based on the properties of the resulting "coumarazines," namely: (1) the difficulty with which they are hydrolysed; (2) their behaviour on oxidation, yielding when $R = H$ or

CH_3 the ring compound $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \text{O} \text{---} \text{CO} \end{smallmatrix}$, whilst, when $\text{R} = \text{C}_6\text{H}_5$, practically no oxidation occurs. In support of the ring formula, moreover, Cebrian cites the inability of *o*-ethoxybenzaldehyde, $\text{EtO} \cdot \text{C}_6\text{H}_4 \cdot \text{CHO}$, to condense with acetamide. On the other hand, he notes the easy solubility of the coumarazines in alkalis, and shows how various salts can be prepared, from which he concludes that "ein aromatisches hydroxyl nachgewiesen ist." By this it is evident that he assumes a phenolic character for the hydroxyl group in the ring

$\text{C}_6\text{H}_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \text{O} \text{---} \text{CR} \cdot \text{OH} \end{smallmatrix}$. This assumption is, however, unwarranted, and,

although there are no exactly similar compounds of certain constitution known for comparison, it can hardly be doubted that a hydroxyl group in such a substance attached to an alkylated carbon atom situated between an oxygen and nitrogen atom would possess secondary or tertiary alcoholic characters rather than phenolic. If this is so the various alkylated and acetylated coumarazines which Cebrian has described are really open-chain compounds of the structure $\text{R}'\text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{CH:N} \cdot \text{CO} \cdot \text{R}$ and $\text{AcO} \cdot \text{C}_6\text{H}_4 \cdot \text{CH:N} \cdot \text{CO} \cdot \text{R}$ respectively.

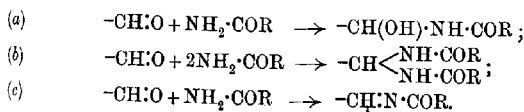
The present authors have not repeated the whole of Cebrian's work, but have confined themselves merely to the condensation which occurs between salicylaldehyde and benzamide, and the results make it clear that not only is no ring compound produced, but also that the amorphous product which Cebrian described as hydroxyphenyl-

coumarazine, $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \text{O} \text{---} \text{CPh} \cdot \text{OH} \end{smallmatrix}$, is a mixture of two open-chain

salicylidenebenzamides, $\text{HO} \cdot \text{C}_6\text{H}_4 \cdot \text{CH:N} \cdot \text{CO} \cdot \text{C}_6\text{H}_5$. Both these substances have the amorphous and other properties attributed to them by Cebrian, but they differ in solubility in acetone, by means of which they were separated. They give the intense purple ferric chloride reaction characteristic of most salicylic derivatives, and possess normal phenolic properties. It is possible that they stand to each other as *syn*- and *anti*-geometrical isomerides, analogous to the corresponding benzylidenebenzylamides (*loc. cit.*), but it is also possible that one is a polymeric modification of the other. Owing to the troublesome amorphous character of the substances, and their lack of special interest, they have not been submitted to further investigation, but in the meantime it may be regarded as certain that, although they may appear in the tautomeric ring-form attributed to them by Cebrian and thus range themselves with the acylsalicylamides as substances exhibiting metoxazone tautomerism, they must be regarded as simple open-chain derivatives, and thus arguments based on the assumed cyclic structure of the "coumarazines" fall through.

It appeared to be of interest incidentally to ascertain whether the true ring compound $C_6H_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \quad \diagdown \\ \text{O} \quad \text{CPh}\cdot\text{OH} \end{smallmatrix}$ could be prepared either from the above compounds or direct from the condensation of salicylaldehyde and benzamide by modifying the conditions; and accordingly a number of experiments have been made, but without success. At the same time, some interesting results have been obtained which show that the same kind of tautomeric change occurs during the process of condensation as occurs in the acylsalicylamide group, resulting in the migration of the benzoyl group (see p. 1937).

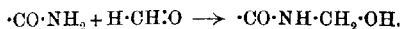
By analogy with the production of phenylbenzometoxazone (*loc. cit.*) in the condensation of salicylamide and benzaldehyde under the influence of hydrogen chloride, it was thought that the above ring compound would be formed from salicylaldehyde and benzamide, using the same catalyst. Carrying out the reaction in benzene solution, hydrogen chloride being passed in, a ready condensation occurs, and a crystalline compound melting at 198° was easily obtained, which gave no ferric chloride reaction, and which at first was assumed to be the desired derivative, $C_6H_4 \begin{smallmatrix} \text{CH:N} \\ \diagup \quad \diagdown \\ \text{O} \quad \text{CPh}\cdot\text{OH} \end{smallmatrix}$, because, on analysis, the values for carbon, hydrogen, and nitrogen agreed with this. A study of the properties of the substance showed, however, that no hydroxyl group was present, and that on cautious hydrolysis a benzoyl group could be eliminated, leaving a compound of phenolic properties still containing benzoyl; whilst a molecular weight determination showed that the value was double that required for the above formula, from which it was evident that the condensation had proceeded on unexpected lines. The references* in the literature to the condensation between amides and aldehydes make it clear that different results may be expected, but there is not sufficient evidence available to correlate these differences with structure of the reacting substances, on the one hand, and conditions of condensation, on the other. In general, the following main types of change have been observed:



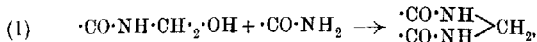
* Roth, *Annalen*, 1870, **154**, 72; Schuster, *Annalen*, 1870, **154**, 80; Medicus, *Londres*, 1871, **157**, 44; Nencki, *Ber.*, 1874, **7**, 158; Hofmann and Meyer, *Ber.*, 1892, **25**, 209; Pulvermacher, *Ber.*, 1892, **25**, 314; Michael and Jeanprêtre, *Ber.*, 1892, **25**, 1678; Bülow, *Ber.*, 1893, **26**, 1972; Cebrian, *Ber.*, 1898, **31**, 1592; Milbourn, *Annalen*, 1905, **343**, 207, 272; Keane and Nicholls, *Trans.*, 1907, **91**, 34; Burrows and Keane, *Trans.*, 1907, **91**, 268; Titherley, *Trans.*, 1907, **91**, 1419.

In all cases the observed results can be explained by assuming an aldol-like condensation (a) to occur first. In restricted cases only, as Einhorn's observations show, can such aldol-like derivatives be isolated, namely, with formaldehyde, chloral, and bromal as aldehydes, or with halogen substituted aliphatic amides.

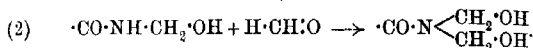
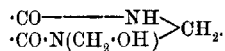
Einhorn has shown that a quite general reaction occurs in presence of alkaline or acid catalysts between formaldehyde, on the one hand, and amides of all kinds, on the other (of monobasic and dibasic aliphatic and aromatic acids), yielding definite aldol-like additive compounds of a type (a), namely, methylol derivatives:



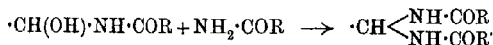
Such methylol derivatives are capable of condensing with (1), a second molecule of amide, or (2) probably with a second molecule of formaldehyde:



which may now condense further with formaldehyde, yielding



Apparently in the case of most amides and aldehydes such aldol-like additive compounds cannot be obtained, but Einhorn's researches have thrown considerable light on the mechanism of the condensation, and there can be little doubt that such hydroxy-derivatives are, in all cases, first formed. In the majority of condensations recorded, further action appears to have occurred between the aldol-compound and a second molecule of amide, giving the type of reaction represented in (b):

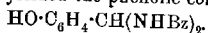


This type of reaction (b) occurs in the following condensations: between benzaldehyde and acetamide, as well as other fatty amides and benzamide (Roth); between anisaldehyde and acetamide, as well as benzamide (Schuster); between benzaldehyde and formamide (Bülow); between heptaldehyde and benzamide (Medicus); between acetaldehyde and carbamide, yielding the compound $\text{CH}_3\cdot\text{CH} \begin{array}{c} \text{NH} \\ \text{NH} \end{array} \text{COO}$ (Nencki)—in each case by simply heating the two substances together; also between acetaldehyde and benzamide (Nencki); between formaldehyde and acetamide in aqueous solution (Pulvermacher); between benzaldehyde and *o*-methoxybenzamide (Keane and Nicholls);

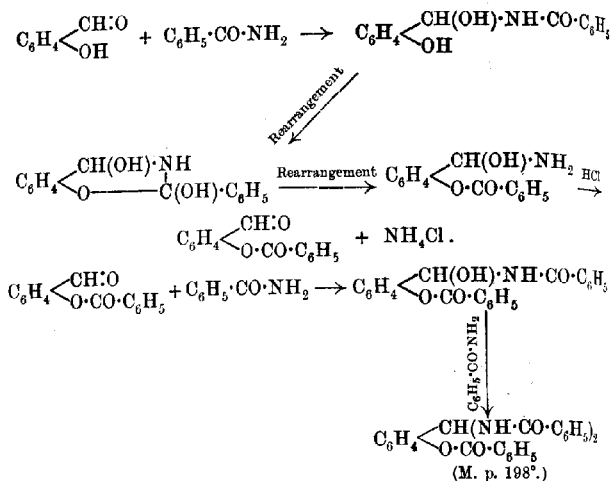
between benzaldehyde and diethylmalonamide, yielding 4:6-diketophenyl-5:5-diethylhexahydropyrimidine, $\text{C}_6\text{H}_5\text{CH}(\text{CO}\cdot\text{NH})_2\text{CH}\cdot\text{C}_6\text{H}_5$ (Burrows and Keane)—in each case in presence of hydrochloric acid as a catalyst; also between formaldehyde and benzamide under the influence of dilute sulphuric acid, producing dibenzoylmethylenediamine, $\text{CH}_2(\text{NHBz})_2$ (Pulvermacher).

The type of change (c) has been observed in the condensation between benzaldehyde and mandelamide simply by heating (Michael and canprêtre), giving the compound $\text{C}_6\text{H}_5\cdot\text{CH}(\text{OH})\cdot\text{CO}\cdot\text{N}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$; between salicylaldehyde and amides in presence of sodium acetate (Lebrian and the authors); between benzaldehyde and salicylamide in presence of sodium acetate or hydrogen chloride (Keane and Nicholls; Metherley), yielding the compound $\text{HO}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{N}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$, and, by rearrangement, the isomeride $\text{C}_6\text{H}_4\cdot\text{O}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$.

From the above condensations, it is impossible to trace fully the influences at play, and predict what would happen in the case of salicylaldehyde and benzamide under the influence of hydrogen chloride. The crystalline derivative obtained was finally proved to be *O-N-N*-tribenzoylsalicylidenediamine, $\text{BzO}\cdot\text{C}_6\text{H}_4\cdot\text{CH}(\text{NHBz})_2$ (M.W. 450), from the fact that on cautious hydrolysis with alcoholic potassium hydroxide it yielded the phenolic compound

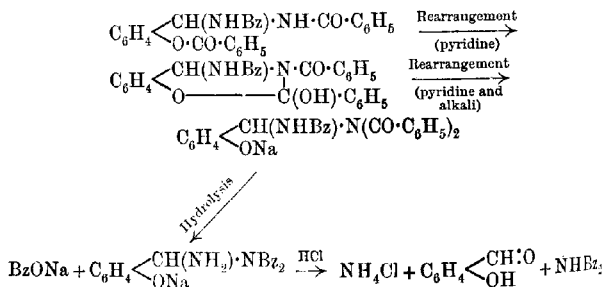


The production of such a tribenzoyl derivative in this unexpected manner furnishes evidence that during the condensation the benzoyl group wanders in a similar manner to that which has been observed by one of the authors in the acylsalicylamide group, and that the same kind of cyclic metoxazone tautomerism is at play. Moreover, when hydrogen chloride acts, not only as a catalyst, but also as a remover of ammonia, which was proved by the fact that a considerable quantity of ammonium chloride is produced, and that the yield is dependent on the continuous passage of the gas, whilst in the preparation of phenylbenzometoxazone by an analogous method a small quantity of hydrogen chloride suffices. The changes which occur in the condensation may be represented thus:



It has been shown, moreover, that a similar wandering of benzoyl occurs when this tribenzoyl derivative is hydrolysed by pyridine and alkali in the cold. Only one benzoyl group is eliminated as benzoic acid, the two others appearing attached to nitrogen in the form of dibenzamide, which is produced together with ammonia and salicyl aldehyde. The production of dibenzamide was so unexpected, having regard to the constitution of the original substance, that it was at first assumed from its analysis to be the desired "coumarazine,"

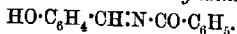
$\text{C}_6\text{H}_4 \begin{array}{l} \text{CH:N} \\ \text{< O} \end{array} \text{---} \text{CPh} \cdot \text{OH}$ which by chance is isomeric with dibenzamide, but close investigation revealed its identity with the latter. The hydrolysis is evidently accompanied by a wandering of benzoyl, and may be expressed thus:



The intermediate amino-derivative, which appeared as an oily mass, could not be isolated, owing to its instability.

EXPERIMENTAL.

Preparation of the Isomeric Salicylidenebenzamides,



Following the method of Cebrian (*loc. cit.*), 10 grams of salicylaldehyde, 10 grams of benzamide, and 6 grams of anhydrous sodium acetate, intimately mixed, were heated at 140° for five hours. The green, semi-solid mass was allowed to cool, and the resulting solid powdered and digested with water. The insoluble, crude material, weighing, after washing and drying, 15 grams, was purified by dissolving in aqueous sodium hydroxide, filtering, and adding acetic acid.

The amorphous, discoloured precipitate, consisting of the two isomerides, was dried and digested three times with an excess of acetone in the cold, by which a nearly complete separation was effected. The insoluble isomeride remained as an amorphous precipitate, which, after washing and drying, was further purified by repeated solution in pyridine and precipitation with acid. The isomeride soluble in acetone was regained from the filtrate by precipitating with faintly acidified water, and purified as far as possible by repeated solution in hot alcohol and precipitation with acidified water. The two compounds, which were produced in approximately equal quantity, have very similar properties.

Salicylidenebenzamide (soluble in acetone) :

0.2024 gave 0.5305 CO_2 and 0.1023 H_2O . $\text{C} = 71.48$; $\text{H} = 5.66$.

0.2304, by Kjeldahl's method, required 9.4 c.c. $N/10$ HCl . $\text{N} = 5.71$.

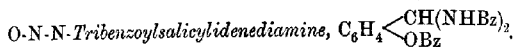
$\text{C}_{14}\text{H}_{11}\text{O}_2\text{N}$ requires $\text{C} = 74.66$; $\text{H} = 4.89$; $\text{N} = 6.22$ per cent.

The substance is a pale yellow powder, which does not melt but darkens at 190° . It is very soluble in cold acetone, fairly so in hot alcohol or pyridine, sparingly so in benzene or chloroform, and insoluble in ether, ethyl acetate, or light petroleum. From solution in hot glacial acetic acid, it separates in a gelatinous form on cooling. In acetone or alcohol solution, it gives a dark purple coloration with ferric chloride, whilst in alkali hydroxides it dissolves readily, yielding a yellow solution, from which it is precipitated in a gelatinous form by acids.

Salicylidenebenzamide (insoluble in acetone):

0.3338 gave 0.9098 CO₂ and 0.1572 H₂O. C = 74.35; H = 5.27.
 0.2337, by Kjeldahl's method, required 10.5 c.c. N/10 HCl. N = 6.28.
 C₁₄H₁₁O₂N requires C = 74.66; H = 4.89; N = 6.22 per cent.

The substance forms a yellow, amorphous powder, which does not melt but decomposes at about 200°. It is soluble in pyridine, but insoluble in chloroform, benzene, alcohol, or ether; it is sparingly soluble in hot acetic acid, and, on cooling, separates with difficulty in a gelatinous form. Its behaviour with acetone is curious; with a little acetone, it mixes to form a homogeneous liquid, doubtless consisting of a solution of acetone in the substance; on adding more acetone, however, the substance at once separates as a thick, flocculent precipitate, which is only very slightly soluble in, and may be washed by, acetone. The substance in presence of acetone gives a strong purple colour with alcoholic ferric chloride. It dissolves at once in alkali hydroxide, forming a yellow solution, from which acids precipitate it in a yellowish-white, gelatinous form.



A mixture of 40 grams of salicylaldehyde and 80 grams of benzamide, dissolved in about 200 c.c. of benzene, was heated to boiling in a reflux apparatus while a steady stream of hydrogen chloride was passed in. The clear solution rapidly became turbid, owing to the formation of water, which also showed itself as drops in the condenser.

At the same time, ammonium chloride continually separated, and collected at the bottom of the flask. After an hour, on cooling, the mass of white crystals which separated was filtered off, washed with benzene, and dried. The mass consisted chiefly of a mixture of the tribenzoyl derivative, benzamide, and ammonium chloride, and the former was separated by boiling the mixture with water and washing thoroughly with hot water. The filtrate, on cooling, deposited benzamide, which was thus recovered. The insoluble tribenzoyl derivative contained small quantities of an amorphous phenolic compound,* which was removed by digestion with 5 per cent. sodium hydroxide.

* This substance was recovered from the alkaline filtrate as a white, gelatinous precipitate by acidifying. Two analyses gave N = 3.48 and 3.45 per cent. It was soluble in alcohol, acetone, pyridine, ethyl acetate, or hot acetic acid, and insoluble in benzene, chloroform, or cold acetic acid. The compound was not further investigated.

The yield was 58 grams, and the product was nearly pure. A further quantity could be obtained from the above benzene filtrate by adding more benzamide and again passing in hydrogen chloride. The substance was purified by crystallisation from hot alcohol, chloroform, ethyl acetate, or methyl alcohol, from which solvents it separates as beautiful long, colourless needles, but the crystallisation from any of these solvents is attended by considerable loss.

0.1763 gave 0.4794 CO_2 and 0.0828 H_2O . $\text{C} = 74.16$; $\text{H} = 5.26$.

0.3663, by Kjeldahl's method, required 16.57 c.c. $\text{N}/10$ HCl . $\text{N} = 6.33$.

0.3165 " " " 14.3 c.c. $\text{N}/10$ HCl . $\text{N} = 6.32$.

0.3760 gave 19.8 c.c. N_2 (moist) at 21° and 762 mm. $\text{N} = 6.01$.

$\text{C}_{25}\text{H}_{22}\text{O}_4\text{N}_2$ requires $\text{C} = 74.66$; $\text{H} = 4.89$; $\text{N} = 6.22$ per cent.

Molecular weight determinations by the ebullioscopic method were made:

0.2893 in 39.5 chloroform gave Δt 0.055° . $\text{M.W.} = 487$.

0.6008 " " " Δt 0.122° . $\text{M.W.} = 456$.

$\text{C}_{25}\text{H}_{22}\text{O}_4\text{N}_2$ requires $\text{M.W.} = 450$.

O-N-N-Tribenzoylsalicylidenediamine melts at 198° ; it is very soluble in pyridine, moderately so in acetone, slightly so in methyl or ethyl alcohol, benzene, acetic acid, ethyl acetate, or chloroform, and insoluble in light petroleum. It is insoluble in dilute acid and alkali, and its acetone solution gives no coloration with alcoholic ferric chloride. It is unaffected by acetyl chloride or benzoyl chloride in presence of pyridine, and in benzene solution by sodium, sodamide, or phosphorus pentachloride.

Hydrolysis of O-N-N-Tribenzoylsalicylidenediamine.

The products of hydrolysis vary with the conditions. By boiling with dilute hydrochloric acid, it is decomposed into salicylaldehyde, benzoic acid, and ammonia, and a similar decomposition is effected by long boiling with aqueous sodium hydroxide.

By hydrolysis in the cold with alkali hydroxide in the presence of pyridine, dibenzamide was produced, as was shown by repeated experiments under varying conditions. In one case the procedure was as follows: Twelve grams of the tribenzoyl derivative were mixed with 12 c.c. of 50 per cent. potassium hydroxide and 2 c.c. of pyridine, and the mixture was well triturated for nearly an hour. An orange-red colour was first produced, which changed to yellow, and the mass was treated with 200 c.c. of water, in which it dissolved to a clear solution. The latter, on acidifying, became milky, and, on extracting with ether, salicylaldehyde and benzoic acid were isolated, whilst the aqueous

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portion, on standing, deposited needles (5.7 grams) which, after recrystallisation from hot water, melted at 146—147°:

0.2346, by Kjeldahl's method, required 10.1 c.c. $N/10$ HCl. $N = 6.03$.

0.2279 gave 12.7 c.c. N_2 (moist) at 26° and 764 mm. $N = 6.22$.

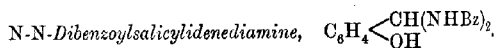
$C_{14}H_{11}O_2N$ requires $N = 6.22$ per cent.

The needles, although colourless, were soluble in sodium hydroxide to a yellow solution, but otherwise possessed all the properties (solubility, &c.) of dibenzamide. Repeated crystallisation from alcohol eliminated the impurity causing the yellow colour, and gave stout prisms, the identity of which with dibenzamide was established (1) by the fact that a mixture with the latter showed no depression in melting point, and (2) by showing that the rate and products (benzamide and benzoic acid) of cautious hydrolysis of the substance in presence of hot alkali were the same as those of dibenzamide.

The production of dibenzamide in the alkali-pyridine hydrolysis of the tribenzoyl derivative is accompanied by formation of ammonia, which was proved thus: The acid liquor containing pyridine, etc., from which the dibenzamide had separated, was evaporated to dryness, the residue extracted while hot with alcohol to remove pyridine hydrochloride, and the insoluble solid was then dissolved in water, rendered alkaline, and distilled into dilute hydrochloric acid, which was then evaporated. About one gram of ammonium chloride was isolated in this way.

In the above alkali-pyridine hydrolysis, there was evidence in several experiments of the presence of an oily intermediate amino-derivative, probably $HO \cdot C_6H_4 \cdot CH(NH_2) \cdot NBz_2$, which appeared to be decomposed by hydrochloric acid, forming ammonium chloride, salicylaldehyde, and dibenzamide.

By the cautious hydrolysis of the tribenzoyl derivative with aqueous alcoholic sodium hydroxide, one benzoyl group may be eliminated forming:



Twelve grams of *O*-*N,N*-tribenzoylsalicylidenediamine were mixed with 50 c.c. of water, 25 c.c. of 10 per cent. aqueous sodium hydroxide, and 150 c.c. of alcohol. The mixture, the colour of which was at first deep red and then changed to yellow, was agitated in the cold continuously until the solid had completely passed into solution, which required about one and a-half hours. The solution was kept cool while very dilute hydrochloric acid was stirred in. The dibenzoyl derivative was obtained in this way in a fairly pure state as a white, amorphous precipitate, which was washed. Ethyl benzoate was formed during the

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hydrolysis, and was isolated by extracting with ether, etc. The precipitate weighed 6.5 grams, melted at 185°, and was further purified by dissolving in a mixture of 10 per cent. aqueous alcoholic sodium hydroxide and reprecipitating cautiously with dilute hydrochloric acid.

The solid, which melted at 192°, was practically pure, and was crystallised from hot methyl or ethyl alcohol or acetone by adding light petroleum gradually and allowing to stand. By either of these methods, which are very wasteful, it was obtained in small, prismatic needles:

0.3777, by Kjeldahl's method, required 21.8 c.c. $N/10$ HCl. $N = 8.08$.
0.1442 gave 9.7 c.c. N_2 (moist) at 20° and 760 mm. $N = 7.69$.

$C_{21}H_{18}O_3N_2$ requires $N = 8.09$ per cent.

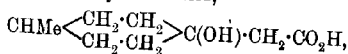
N,N-Dibenzoylsalicylidenediamine is soluble in cold pyridine, acetone, or alcohol, or acetic acid, and is insoluble in ether, benzene, chloroform, or light petroleum. It is only very slowly soluble in cold dilute aqueous sodium hydroxide, forming a light yellow, sparingly soluble sodium salt, but it is much more readily soluble in alkali in presence of aqueous alcohol. In acetone solution it gives a characteristic green coloration with alcoholic ferric chloride, and the colour changes purple on heating, owing apparently to decomposition.

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UNIVERSITY OF LIVERPOOL.

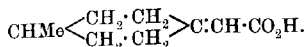
XCIV.—Experiments on the Synthesis of 1-Methylcyclohexylidene-4-acetic Acid. Part II.

VICTOR JOHN HARDING, WALTER NORMAN HAWORTH, and
WILLIAM HENRY PERKIN, jun.

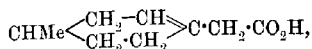
Part I of this investigation (Trans., 1908, 93, 1075) a series of experiments was described which was undertaken with the object of comparing the above acid, and of ascertaining whether it could be converted into active modifications by the crystallisation of its salts with optically active bases. It was pointed out that, while these experiments were in progress, W. Marckwald and R. Meth (*Ber.*, 1906, 39, 1171) were engaged with the same problem, and had prepared an acid (m. p. 41°) by the elimination of water from hydroxy-1-methylcyclohexylacetic acid,



which they pronounced to be 1-methylcyclohexylidene-4-acetic acid,



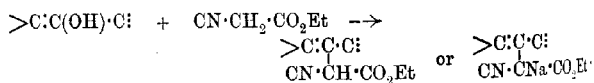
They also showed that the acid may be resolved into its *d*- and *l*-modifications by the fractional crystallisation of the cinchonine salts. In subsequent communications (*ibid.*, 2035 and 2404) they described further experiments, which, in their opinion, clearly demonstrated that their view of the constitution of this acid was the correct one. On the other hand, Perkin and Pope (*loc. cit.*) pointed out that the elimination of water from 4-hydroxy-1-methylcyclohexylacetic acid may take place in a different direction from that assumed by Marckwald and Meth, and may lead to the formation of 1-methyl- Δ^3 -cyclohexene-4-acetic acid,



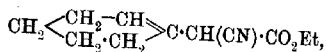
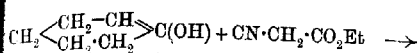
and they expressed the opinion that this formula most probably represents the constitution of the acid of melting point 41° .

If this view should prove to be correct, the acid would contain an asymmetric carbon atom, and there would then be nothing remarkable in its resolution into active modifications in the manner described by Marckwald and Meth. During the last two years, we have been engaged in a detailed investigation of the difficult problem of the constitution of this acid, and have obtained experimental evidence which we think proves without doubt that the acid of melting point 41° is 1-methyl- Δ^3 -cyclohexene-4-acetic acid, and that the view of Marckwald and Meth that this acid is 1-methylcyclohexylidene-4-acetic acid cannot be accepted as correct.

The first indirect evidence of constitution was obtained during the course of a series of researches, which we hope shortly to publish, on the condensation of ketones with ethyl cyanoacetate in the presence of sodium ethoxide or piperidine. The reaction takes place in such a way that the enolic modification of the ketone condenses with the ethyl cyanoacetate (or cyanoacetic acid) with elimination of water, and the result is either the free condensation product or its sodium derivative, according as to whether piperidine or sodium ethoxide has been employed:

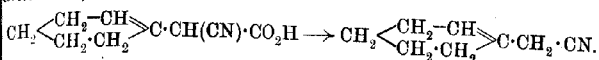


When this interesting reaction was investigated in the case of cyclohexanone, it was found that condensation took place readily, and that elimination of water proceeded almost entirely in the direction:



that is to say, with the production of *ethyl α-cyano-Δ¹-cyclohexeneacetate*.

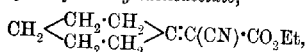
This cyano-ester is readily hydrolysed with formation of *α-cyano-Δ¹-cyclohexeneacetic acid*, and this, on distillation, is decomposed with elimination of carbon dioxide and formation of *Δ¹-cyclohexeneacetonitrile*,



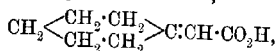
When this nitrile is hydrolysed under the conditions described in detail in the experimental part (p. 1959), it yields *Δ¹-cyclohexeneacetic acid*,



which melts at 38°. Whilst then the condensation of *cyclohexanone* with ethyl cyanoacetate proceeds almost entirely in the direction described above, the product nevertheless contains small quantities of the isomeric *ethyl cyanocyclohexylideneacetate*,

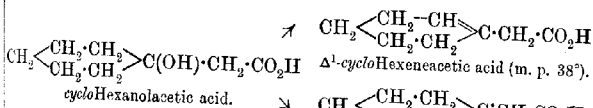


and from this a small quantity of a nitrile is obtained which, on hydrolysis, yields *cyclohexylideneacetic acid*,



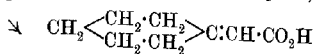
which melts at 91°.

These acids of melting points 38° and 91° are identical with the acids which Wallach (*Annalen*, 1907, 353, 292) obtained by the elimination of water from *cyclohexanolacetic acid*:



Δ¹-cyclohexeneacetic acid (m. p. 38°).

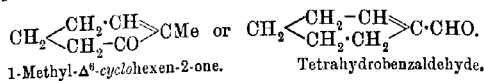
cyclohexanolacetic acid.



cyclohexylideneacetic acid (m. p. 91°).

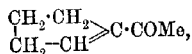
As in the cyanoacetic ester and *cyclohexanone* condensation just described, so also during the decomposition of *cyclohexanolacetic acid*, the unsaturated acid produced in by far the larger quantity is *Δ¹-cyclohexeneacetic acid* (m. p. 38°). The question of the constitution of these acids has been very carefully investigated by Wallach (*loc. cit.*). The acid of melting point 91° yields *cyclohexanone* when it is oxidised with permanganate, and it is therefore evidently

cyclohexylidene-4-acetic acid. The nature of the change which takes place when the acid of melting point 38° is oxidised by permanganate has proved to be much more difficult to understand. The product of oxidation contains a volatile oil, $C_7H_{10}O$, the semicarbazone of which has the formula $C_8H_{13}N_3O$ and the melting point $203-204^\circ$, and the oil, on oxidation with chromic acid, yields glutaric acid. Wallach at first expressed the opinion that the substance $C_7H_{10}O$ was either



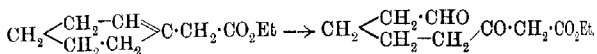
In a later paper (*Annalen*, 1908, 359, 310), he shows that this substance is neither 1-methyl- Δ^1 -cyclohexen-2-one nor tetrahydrobenzaldehyde, and suggests that its constitution is possibly represented by the formula $\text{CH}_2 \begin{array}{c} \text{CH}_2 \cdot \text{CH}_2 \\ \text{CH}_2 \cdot \text{CO} \end{array} \text{C} \cdot \text{CH}_2$.

Experiments on the oxidation of the ester of the acid of melting point 38° , which are described below, clearly indicate, however, that the substance $C_7H_{10}O$ is Δ^1 -cyclopentene methyl ketone,

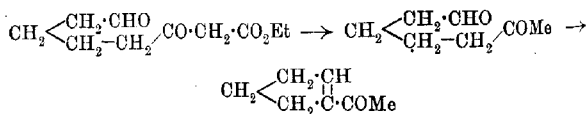


and this view is in harmony with the fact that this ketone yields glutaric acid when it is oxidised with chromic acid.

When ethyl Δ^1 -cyclohexeneacetate is oxidised by ozone in chloroform solution in the presence of water, it is converted into an ester, which gives in alcoholic solution with ferric chloride a violet coloration, and the formation of which may be doubtless expressed thus:

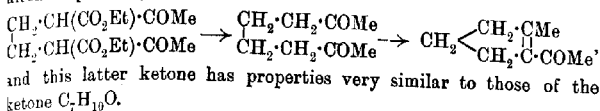


This ester is readily decomposed by boiling dilute sulphuric acid with elimination of alcohol and carbon dioxide and formation of a ketone, $C_7H_{10}O$, which yields a semicarbazone of melting point $203-204^\circ$, and is evidently identical with the ketone possessing these properties which Wallach prepared in the manner just described. We have no doubt that this ketone is Δ^1 -cyclopentene methyl ketone, and that its formation is due to the following series of changes.



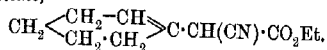
Some years ago (*Trans.*, 1890, 57, 242), Marshall and Perkin prepared a methyl derivative of this ketone by a very similar

condensation, namely, from ethyl diacetyladipate by the action of alcoholic potash.

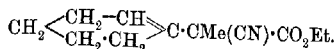


and this latter ketone has properties very similar to those of the ketone $\text{C}_7\text{H}_{10}\text{O}$.

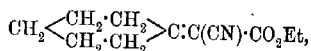
When the method of formation and properties of the acid of melting point 38° , its behaviour on oxidation, and its isomerism with *cyclohexylideneacetic acid*, m. p. 91° , are taken into account, there can no longer be any doubt that Wallach's view that it is Δ^1 -*cyclohexeneacetic acid* must be accepted as correct. But this acid is practically the sole product of the hydrolysis of the ester which is produced when *cyclohexanone* is condensed with ethyl cyanoacetate (p. 1945), and it follows therefore that this ester must consist almost entirely of *ethyl α -cyano- Δ^1 -cyclohexeneacetate*,



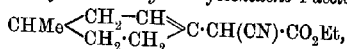
This point is, however, so important that we confirmed it by investigating the action of sodium and methyl iodide on the cyano-ester, and obtained an almost quantitative yield of *ethyl α -cyano- α - Δ^1 -cyclohexene- γ -proionate*,



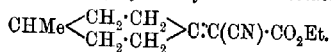
The description of this substance and of its decomposition products, and also of the analogous derivative obtained with the aid of ω -bromocetophenone, will be found on pp. 1958 and 1961. The formation of these substitution products can only be explained on the assumption that the original ester contains the grouping $-\text{CH}(\text{CN})\cdot\text{CO}_2\text{Et}$; the isomeric ester,



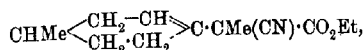
which might, conceivably, result from the condensation of *cyclohexanone* with ethyl cyanoacetate, does not contain a hydrogen atom replaceable by sodium. We now investigated the action between *1-methylcyclohexan-4-one* and ethyl cyanoacetate in the presence of sodium ethoxide or piperidine, and found that the condensation evidently proceeded in a similar manner to that already described in the case of *cyclohexanone*, and there was no reason to doubt that the product was *ethyl α -cyano-1-methyl- Δ^3 -cyclohexene-4-acetate*,



and not *ethyl α -cyano-1-methylcyclohexylidene-4-acetate*,

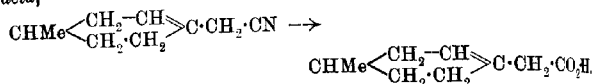


This view was, however, controlled by investigating the behaviour of the ester with sodium and methyl iodide (p. 1972), when an almost quantitative yield of *ethyl- α -cyano- α -1-methyl- Δ^3 -cyclohexene-4-propionate*,

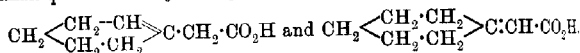


was obtained, and a similar substitution product resulted from the action of bromoacetophenone on the sodium derivative (p. 1966).

Ethyl α -cyano-1-methyl- Δ^3 -cyclohexene-4-acetate is readily hydrolysed, and the free acid is decomposed on distillation with elimination of carbon dioxide and formation of 1-methyl- Δ^3 -cyclohexene-4-acetonitrile. When this nitrile is digested with alcohol and sulphuric acid and the resulting ester hydrolysed, it yields 1-methyl- Δ^3 -cyclohexene-4-acetic acid,



The acid obtained in this way melted at 41° , and was found by direct comparison to be identical with the acid described by Marckwald and Meth (*Ber.*, 1906, **39**, 1171) as 1-methylcyclohexylidene-4-acetic acid. During the investigation of the condensation of cyclohexanone and ethyl cyanoacetate (p. 1945), we were able to isolate from the product, not only Δ^1 -cyclohexeneacetic acid (m. p. 38°), but also very small quantities of cyclohexylideneacetic acid (m. p. 91°):



In the present case, however, 1-methyl- Δ^3 -cyclohexene-4-acetic acid appears to be the only acid formed, since, in spite of careful search, we were unable to isolate even traces of 1-methylcyclohexylidene-4-acetic acid,

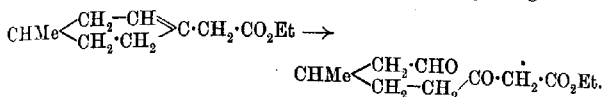


Whilst, then, the experiments described above seem to us to be very strong evidence that the acid of Marckwald and Meth (m. p. 41°) is 1-methyl- Δ^3 -cyclohexene-4-acetic acid, we have carried out a further investigation of this acid with the view, not only to emphasise this point, but also clearly to establish the fact that this acid is in all respects the counterpart of Wallach's Δ^1 -cyclohexeneacetic acid (m. p. 38°). Probably the most characteristic property of Δ^1 -cyclohexeneacetic acid is the behaviour of its ester with ozone (p. 1946), and we have found that, under the same conditions, 1-methyl- Δ^3 -cyclohexene-4-acetic acid (m. p. 41°) behaves in an exactly similar manner. When the ester of this acid is oxidised with ozone in chloroform solution and in the presence of water, it is converted into an ester which

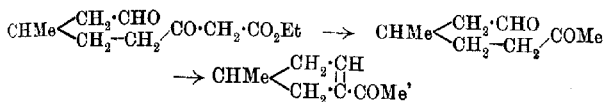
SYNTHESIS OF 1-METHYLCYCLOHEXYLIDENE-4-ACETIC ACID. 1949

yields an intense violet coloration when ferric chloride is added to its alcoholic solution.

There can be little doubt that this ester is *ethyl ζ-aldehydo-β-keto-isooctate*, and that its formation is due to the following change :

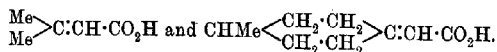


When this ester is digested with dilute sulphuric acid, it is decomposed with elimination of alcohol and carbon dioxide and formation of a ketone, $\text{C}_8\text{H}_{12}\text{O}$, which yields a semicarbazone (m. p. 207–209°), and is obviously a simple homologue of the ketone, $\text{C}_7\text{H}_{10}\text{O}$, obtained under similar conditions from *cyclohexeneacetic acid* (m. p. 38° : compare p. 1946). The formation of this ketone is readily understood from the following scheme :

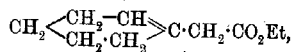


and the ketone, $\text{C}_8\text{H}_{12}\text{O}$, is therefore 1-methyl- Δ^3 -4-cyclopentene methyl ketone.

While these experiments were in progress, we were struck with the similarity in structure between $\beta\beta$ -dimethylacrylic acid and 1-methylcyclohexylidene-4-acetic acid :



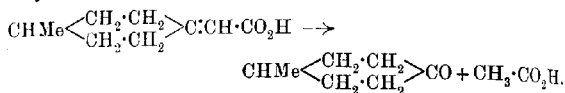
Now it is well known that ethyl $\beta\beta$ -dimethylacrylate, like all esters containing the group $>\text{C} \cdot \text{CH} \cdot \text{CO}_2\text{Et}$, readily condenses with ethyl sodiocyanoacetate with formation of the sodium derivative of ethyl cyanodimethylglutarate, $\text{CO}_2\text{Et} \cdot \text{C}(\text{CN})\text{Na} \cdot \text{CMe}_2 \cdot \text{CH}_2 \cdot \text{CO}_2\text{Et}$ (Perkin and Thorpe, *Trans.*, 1899, 75, 901). Since this reaction is not shown by acids like 1-methyl- Δ^3 -cyclohexene-4-acetic acid, which contain the double linking in the $\beta\gamma$ -position, it was thought that valuable evidence of structure might be obtained by employing it in connexion with the present investigation. We therefore digested the ester of the acid of melting point 41° with ethyl sodiocyanoacetate for several hours, but no additive compound was produced, and practically the whole of the acid was recovered unchanged. When the same experiment was made with ethyl Δ^1 -cyclohexeneacetate,



which admittedly contains the double linking in the $\beta\gamma$ -position, an exactly similar result was obtained, and no trace of an additive com-

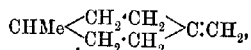
pound was produced. This experiment is not only strong evidence that the acid of melting point 41° cannot be 1-methylcyclohexylidene-4-acetic acid, but it is also a further example of the striking similarity in properties between this acid and Δ^1 -cyclohexeneacetic acid.

We next examined in detail the evidence brought forward by Marckwald and Meth in support of their contention that the acid (m. p. 41°) is 1-methylcyclohexylidene-4-acetic acid. In the first place these investigators (*Ber.*, 1906, 39, 1175) state that the acid yields methylcyclohexanone and potassium acetate when it is heated with potassium hydroxide and a little water, a change which they obviously assume to be due to disruption at the double linking by hydrolysis:



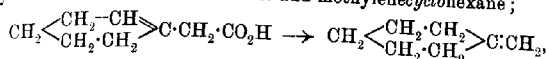
We have repeated the experiment and can confirm the result, but we find that, in order to bring about this change, the acid has practically to be fused with potassium hydroxide. The acid may be heated at 210° with 90 per cent. aqueous potassium hydroxide without any apparent change, but, when distilled with potassium hydroxide and a trace of water, it is ultimately decomposed with formation of methylcyclohexanone and potassium acetate. Perkin and Pope (*Proc.*, 1906, 22, 108) have already pointed out that it is well known that $\beta\gamma$ -unsaturated acids, like 1-methyl- Δ^3 -cyclohexene-4-acetic acid are, not infrequently, converted by treatment with strong alkali into $\alpha\beta$ -unsaturated acids, and that this isomeric change may have taken place in the case of Marckwald and Meth's acid prior to the decomposition into methylcyclohexanone and acetic acid; in a later paper (*Ber.*, 1906, 39, 2036), Marckwald and Meth acknowledge the justice of this criticism. This behaviour with potassium hydroxide is, in fact, strong evidence in support of our view that the acid is 1-methyl- Δ^3 -cyclohexene-4-acetic acid, since, if it were 1-methylcyclohexylidene-4-acetic acid, it might be expected to yield methylcyclohexanone when boiled with strong aqueous potassium hydroxide much more readily than is actually the case.

In their second communication (*Ber.*, 1906, 39, 2036), Marckwald and Meth show that the acid of melting point 41° is decomposed on distillation with elimination of carbon dioxide and formation of a hydrocarbon, which, as they suggest, is doubtless 1-methyl-4-methylene-cyclohexane,

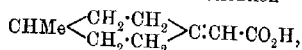


and they regard this as evidence that the double linking in the acid

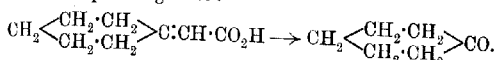
must be in the side-chain, and that therefore the acid must be 1-methylcyclohexylidene-4-acetic acid. But Wallach (*Annalen*, 1908, 360, 27) has shown that Δ^1 -cyclohexeneacetic acid is decomposed by dry distillation into carbon dioxide and methylenecyclohexane;



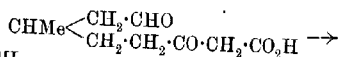
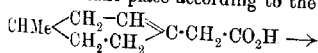
and he makes the general statement that, when acids of this type are distilled, there is a tendency for the double linking to change from the ring to the side-chain. It follows therefore that the behaviour of 1-methyl- Δ^1 -cyclohexene-4-acetic acid in this respect is quite normal, and that the formation of 1-methyl-4-methylenecyclohexane by distillation cannot be used as an argument in favour of the view of Marckwald and Meth that this acid is 1-methylcyclohexylidene-4-acetic acid. Very strong evidence against this view is the behaviour of this acid (m. p. 41°) on oxidation with permanganate. If this acid has the constitution



it should behave, on oxidation, like other acids containing the group $>\text{C} \cdot \text{CH} \cdot \text{CO}_2\text{H}$, that is to say, it should yield the corresponding aldehyde (or ketone) and oxalic acid. In agreement with this view is the observation of Wallach (*Annalen*, 1907, 353, 289), that cyclohexylideneacetic acid (m. p. 91°) readily yields cyclohexanone when it is oxidised with permanganate:

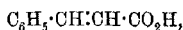


But Marckwald and Meth (*Ber.*, 1906, 39, 2037) specially mention that they were unable to obtain 1-methylcyclohexan-4-one from the acid of melting point 41° by oxidation, and, although we have investigated the behaviour of this acid towards permanganate under a variety of conditions, we have never succeeded in demonstrating the formation even of a trace of methylcyclohexanone. We cannot therefore confirm the statement of Wallach and Evans (*Annalen*, 1907, 353, 311), that methylcyclohexanone is produced when the acid (m. p. 41°) is oxidised by permanganate, and are of opinion that the ketone which these investigators obtained was 1-methyl- Δ^3 -4-cyclopentene methyl ketone, the formation of which may well be assumed to take place according to the following scheme:

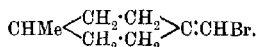


This process is, in fact, very similar to that which is known to take place when Δ^1 -cyclohexeneacetic acid is oxidised by permanganate (p. 1946), or when its ester or the ester of 1-methyl- Δ^3 -cyclohexene-4-acetic acid is treated with ozone (compare p. 1946).

In their last communication on 1-methylcyclohexylidene-4-acetic acid (*Ber.*, 1908, 39, 2405), Marckwald and Meth deduce fresh evidence which they state removes any possible remaining doubt that the acid of melting point 41° is 1-methylcyclohexylidene-4-acetic acid. They point out quite correctly * that cinnamic acid,



is decomposed when it is treated with sodium carbonate and bromine water with separation of ω -bromostyrene, $C_6H_5 \cdot CH:CHBr$, and state that their acid of melting point 41° under the same conditions and in the cold yields in an exactly analogous manner the bromohydrocarbon,



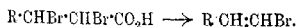
If this were correct, this experimental evidence would no doubt strongly support their view that the acid of melting point 41° is 1-methylcyclohexylidene-4-acetic acid. But Marckwald and Meth have been deceived by appearances. It is true that the addition of bromine water to the cold solution of the acid in sodium carbonate causes the immediate separation of a heavy oil, just as in the case of cinnamic acid; *but this oil is not a bromohydrocarbon.*

If the product is left in a cool place, or, better, extracted with ether, the oil soon solidifies, and, after crystallisation, melts at 103° .

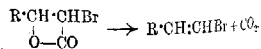
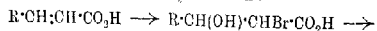
Analysis shows that this beautiful substance has the formula $C_9H_{13}O_2Br$, and is a bromo-lactone.

That it is a lactone is shown by the fact that, whilst insoluble in cold sodium carbonate, it dissolves in warm dilute potassium hydroxide, and, when digested with excess of alkali hydroxide, it is converted into

* Except that the conversion of an unsaturated acid into the bromohydrocarbon by treatment in sodium carbonate solution with bromine water, surely does not take place in the way they represent, namely,

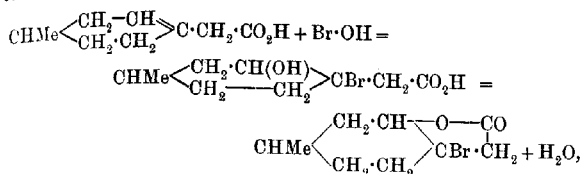


The probable course of this important change seems to be the addition, in the first place, of hypobromous acid to the double linking, followed by the formation of the bromo β -lactone, which then decomposes with elimination of carbon dioxide and separation of the bromohydrocarbon:



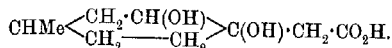
SYNTHESIS OF 1-METHYLCYCLOHEXYLIDENE-4-ACETIC ACID. 1953

the corresponding dihydroxy-acid, $C_8H_{18}(OH)_2 \cdot CO_2H$. The formation of the bromo-lactone is readily understood from the scheme: *



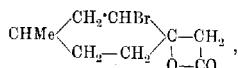
and is in strict accordance with the view that the acid of melting point 41° is 1-methyl- Δ^3 -cyclohexene-4-acetic acid.

The dihydroxy-acid obtained by hydrolysis with potassium hydroxide is doubtless 3:4-dihydroxy-1-methylcyclohexane-4-acetic acid,

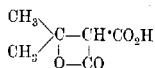


Marckwald and Meth (*loc. cit.*) claim to have proved that the product of the action of bromine water and sodium carbonate on the acid of melting point 41° is 4-bromo-1-methylmethylenecyclohexane (compare p. 1952) by studying its behaviour with water at $140-150^\circ$.

* This bromo-lactone may, of course, be a β -lactone of the formula

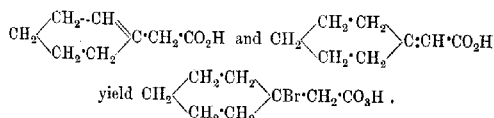


and, when the similarity in structure between this substance and the β -lactone of β -hydroxyisopropylmalonic acid,

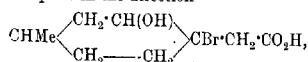


(Meldrum, *Trans.*, 1908, **93**, 598), is taken into account, this view, although improbable, is not at all impossible.

Wallach (*Annalen*, 1907, **353**, 289) has, however, shown that both Δ^3 -cyclohexene-acetic acid and cyclohexylideneacetic acid yield the same bromo-acid on treatment with hydrogen bromide:

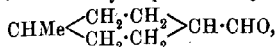


It is therefore probable that the addition of hypobromous acid to 1-methyl- Δ^3 -cyclohexene-4-acetic acid takes place in the direction



in which case a bromo- γ -lactone would result.

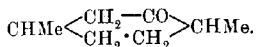
They obtained in this way a substance, the semicarbazone of which corresponded with that of hexahydro-*p*-tolualdehyde,



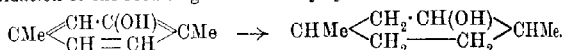
and they therefore concluded that this substance had been produced.

Wallach (*Annalen*, 1906, **347**, 346), who first prepared this aldehyde in small quantities, states that the melting point of a specimen of the semicarbazone after crystallisation from water was 154–156°.

As the formation of this aldehyde was not in accord with the constitution ascribed by us to the bromo-lactone, which is the actual product of the action of bromine and sodium carbonate on the acid of melting point 41°, we heated this lactone with water at 140–150°, and found that it was converted almost quantitatively into a *ketone*, which yielded a semicarbazone of melting point 167°. We were ultimately able to prove that this ketone is 1:4-dimethylcyclohexan-2-one,

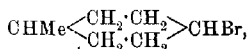


This ketone has recently been described by Sabatier and Mailhe (*Compt. rend.*, 1906, **142**, 553), who obtained it from *p*-xylenol by reduction with hydrogen in the presence of nickel and subsequent oxidation of the resulting 1:4-dimethylcyclohexan-2-ol:



We have prepared a pure specimen of the ketone (b. p. 176°) by this method, and converted it into the semicarbazone, which melted at 155°, but, after several crystallisations from methyl alcohol, the melting point rose to 167°. On comparing this ketone with the ketone which we had obtained from the bromo-lactone by the action of water at 140–150°, it was at once clear that the two specimens were identical. Thus, for example, when the semicarbazones were mixed, there was no alteration in melting point. There can be little doubt that the substance which Marekwald and Meth described as hexahydro-*p*-tolualdehyde was, in reality, 1:4-dimethylcyclohexan-2-one, and the low melting point which they appear to have observed for the semicarbazone was due to that derivative not being quite pure.

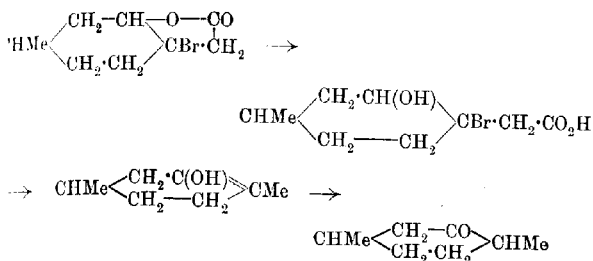
In order to be still more certain on this point, we have prepared hexahydro-*p*-tolualdehyde by a new method, namely, from 4-bromo-1-methylcyclohexane,



by treatment with magnesium and orthoformic ester (p. 1974).

The semicarbazone melted, after crystallisation from dilute methyl alcohol, at 154° , as stated by Wallach (*Annalen*, 1906, **347**, 346), and, when this was mixed with the semicarbazone of the substance produced from the bromo-lactone by the action of water at $140-150^{\circ}$, the mixture melted at about 138° .

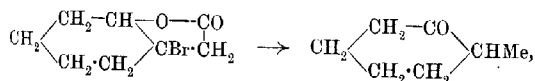
The formation of 1:4-dimethylcyclohexan-2-one from the bromo-lactone by the action of water at $140-150^{\circ}$ is readily explained by the following series of changes:



and the production of 1:4-dimethylcyclohexan-2-one in this way is gain strong evidence in favour of the view that the acid of melting point 41° is 1-methyl- Δ^3 -cyclohexene-4-acetic acid.

In order to demonstrate, in this respect also, the complete analogy between this acid and Δ^1 -cyclohexeneacetic acid (m. p. 38°), we have submitted the latter to the action of sodium carbonate and bromine under exactly the same conditions and with a precisely similar result.

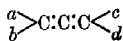
The addition of bromine water to the solution of this acid in sodium carbonate causes the immediate separation of an oil, which crystallises with difficulty, melts at about 70° , and is a bromo-lactone of the formula $\text{C}_8\text{H}_{13}\text{O}_2\text{Br}$. When this bromo-lactone is heated with water at $140-150^{\circ}$, it is decomposed with formation of 1-methylcyclohexan-2-one:



behaviour which is exactly analogous to the conversion of the bromo-lactone, $\text{C}_8\text{H}_{13}\text{O}_2\text{Br}$, from the acid of melting point 41° into 1:4-dimethylcyclohexan-2-one.

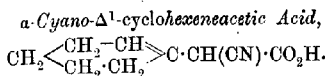
It appears to us that the above experimental evidence proves conclusively that the acid of melting point 41° is 1-methyl- Δ^3 -cyclohexene-4-acetic acid, and that the view of Marckwald and Meth that it is 1-methylcyclohexylidene-4-acetic acid must be abandoned.

It follows therefore that, in spite of the interesting and valuable work of these investigators, the problem of the resolution of a substance of the type



(compare Trans., 1908, 93, 1075) still awaits solution.

EXPERIMENTAL.



In preparing ethyl *α*-cyano-*Δ*¹-cyclohexeneacetate, ethyl cyanoacetate (56 grams) was added to a solution of sodium (12 grams) in alcohol, and, after heating to boiling, cyclohexanone (54 grams) was added; as soon as the sodium derivative had passed into solution, the product was rapidly cooled, diluted with water, acidified with hydrochloric acid, and extracted with ether. The ethereal solution was washed with dilute sodium carbonate (A), dried and evaporated, and the residue distilled under reduced pressure, when, after some unchanged cyclohexanone had passed over, ethyl *α*-cyano-*Δ*¹-cyclohexeneacetate (15 grams) distilled constantly at 174°/25 mm. as a viscous oil possessing the odour of a nitrile:

0.1698 gave 10.8 c.c. N₂ at 18° and 774 mm. N = 7.5.

C₁₁H₁₅O₂N requires N = 7.2 per cent.

When the mixture of the sodium derivative of ethyl cyanoacetate and cyclohexanone (prepared as described above) is boiled on the water-bath for an hour, it becomes reddish-brown, and a sodium salt separates.

If the product is decomposed with acid and the ethereal extract treated with sodium carbonate, it yields a neutral oil and an acid. The neutral oil contains cyclohexanone, *Δ*¹-cyclohexeneacetoneitrile (p. 1959), and a small quantity of ethyl *α*-cyano-*Δ*¹-cyclohexeneacetate.

The sodium carbonate extract yields, on acidifying, a viscous syrup, which, when cooled in ice and salt and stirred, soon solidifies, and, in contact with porous porcelain, the oily impurity is gradually absorbed, leaving a solid residue of crude *α*-cyano-*Δ*¹-cyclohexeneacetic acid, which is best purified by recrystallisation from benzene:

0.1302 gave 0.3096 CO₂ and 0.0760 H₂O. C = 65.6; H = 6.4.

0.1502 „ 11.4 c.c. N₂ at 21° and 775 mm. N = 8.5.

C₉H₁₁O₂N requires C = 65.5; H = 6.6; N = 8.5 per cent.

α-Cyano-*Δ*¹-cyclohexeneacetic acid separates from benzene in short needles, and melts at 109–110°. It is almost insoluble in cold

at more readily so in hot, water, and crystallises, on cooling, in regular groups of needles. It does not react with hydrobromic acid in the cold, but its solution in sodium carbonate instantly decolorises permanganate. Its solution in strong potassium hydroxide gradually decomposes with separation of cyclohexanone.

When the sodium carbonate extract (A) (p. 1956) is acidified and extracted with ether, a yellow syrupy acid is obtained in quantity, which, on long standing, partly crystallises. This was left in contact with porous porcelain until free from oil, and recrystallised either from water or by adding light petroleum to its solution in ether:

I. 0.1143 gave 0.2376 CO_2 and 0.0691 H_2O . $\text{C} = 56.6$; $\text{H} = 6.7$.

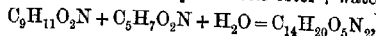
II. 0.1125 „ 0.2330 CO_2 „ 0.0706 H_2O . $\text{C} = 56.5$; $\text{H} = 6.9$.

III. 0.1199 „ 10.2 c.c. N_2 at 12° and 753 mm. $\text{N} = 9.5$.

IV. 0.1575 „ 13.2 c.c. N_2 „ 17° „ 752 mm. $\text{N} = 9.6$.

$\text{C}_{14}\text{H}_{20}\text{O}_5\text{N}_2$ requires $\text{C} = 56.8$; $\text{H} = 6.8$; $\text{N} = 9.4$ per cent.

Analyses I and III were made with substance which had been crystallised from water; II and IV from ether and light petroleum. The basicity of this acid was determined by titration with decinormal sodium hydroxide, when 0.2441 neutralised 0.0330 NaOH , whereas this amount of a monobasic acid, $\text{C}_{14}\text{H}_{20}\text{O}_5\text{N}_2$, should neutralise 0.0340 NaOH . This curious acid has the composition of Δ^1 -cyclohexeneacetic acid + cyanoacetic ester + water:



It was obtained on several occasions and always with the same properties, so that it is doubtless a definite substance.

When it is boiled with sodium carbonate, it is decomposed into cyano- Δ^1 -cyclohexeneacetic acid and an oil, which is evidently cyanoacetic ester. It is difficult to assign a satisfactory constitutional formula to this acid.

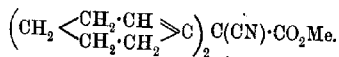
During the course of an experiment in which cyclohexanone and methyl cyanoacetate were condensed in methyl-alcoholic solution, the resulting crude methyl α -cyano- Δ^1 -cyclohexeneacetate gradually deposited a small quantity of a crystalline substance. This was collected and crystallised from benzene, from which it separated in short prisms melting at 170 — 171° :

0.1366 gave 0.3724 CO_2 and 0.0971 H_2O . $\text{C} = 74.4$; $\text{H} = 7.9$.

0.1395 „ 7.0 c.c. N_2 at 16° and 755 mm. $\text{N} = 5.8$.

$\text{C}_{16}\text{H}_{21}\text{O}_2\text{N}$ requires $\text{C} = 74.1$; $\text{H} = 8.1$; $\text{N} = 5.4$ per cent.

This substance is evidently methyl α -cyanodi- Δ^1 -cyclohexeneacetate, and doubtless has the constitution



It was not further investigated.

The Condensation of cyclohexanone with Ethyl Cyanoacetate and with Cyanoacetic Acid in the Presence of Piperidine.

When equimolecular proportions of *cyclohexanone* and *ethyl cyanoacetate* are mixed with a few drops of *piperidine* and allowed to stand, condensation takes place readily with separation of water.

The product is heated for two hours on the water-bath, diluted with water, and extracted with ether, the ethereal solution well washed with dilute hydrochloric acid, dried and evaporated, and the pale yellow oil distilled under reduced pressure, when a good yield of *ethyl α-cyano-Δ¹-cyclohexeneacetate* (p. 1956), distilling at 165–167°/15 mm., is obtained:

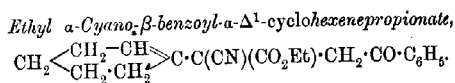
0.1393 gave 0.3463 CO₂ and 0.0947 H₂O. C = 67.9; H = 7.7.

0.1836 „ 12.2 c.c. N₂ at 15° and 754 mm. N = 7.7.

C₁₁H₁₅O₂N requires C = 68.4; H = 7.8; N = 7.2 per cent.

α-Cyano-Δ¹-cyclohexeneacetic acid is readily obtained directly when *cyclohexanone* (7 grams) is added to a mixture of *cyanoacetic acid* (6 grams) and *piperidine* (8 grams). The mixture is allowed to stand for two hours, and then heated on the water-bath for one hour.

After diluting with water, the product acidified, is extracted with ether, the ethereal solution well shaken with sodium carbonate, the alkaline solution acidified, and again extracted with ether. The ethereal extract deposits, on evaporation, a good yield of crude *α-cyano-Δ¹-cyclohexeneacetic acid*, which, after crystallisation from benzene, melts at 109–110°.



This crystalline substance was prepared with the object of proving the identity of the oily esters (*ethyl α-cyano-Δ¹-cyclohexeneacetate*) obtained by the condensation of *cyclohexanone* with *ethyl cyanoacetate* in the presence of sodium and *piperidine* respectively (compare p. 1956 and the last section). In each case, the ester (10 grams) was mixed with sodium (1.2 grams), dissolved in alcohol, and then *α-bromocyclohexenone* (10 grams) gradually added to the yellow solution.

Decomposition commences in the cold, and is completed by heating on the water-bath for fifteen minutes. The product is diluted with water, extracted with ether, the ethereal solution washed well, and evaporated, when a syrup remains, which, if rubbed under water with a sharp glass rod, gradually crystallises. The mass was left in contact with porous porcelain until quite hard, and then crystallised twice.

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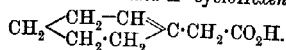
from light petroleum, from which it separated in crusts melting at 7°.

0.1560 gave 6.4 c.c. N_2 at 24° and 760 mm. $N = 4.6$.

$C_{19}H_{21}O_3N$ requires $N = 4.5$.

Experiments showed that the derivatives which resulted in the above manner from the cyano-esters, obtained by the sodium and piperidine condensations, were identical in all respects.

Δ^1 -cycloHexeneacetonitrile and Δ^1 -cycloHexeneacetic Acid,



α -Cyano- Δ^1 -cyclohexeneacetic acid is readily decomposed on slow distillation under a pressure of 90 mm., with elimination of carbon oxide and formation of Δ^1 -cyclohexeneacetonitrile, which distils at about 120–160° in the first instance. The temperature then rises rapidly, and, if the distillation is continued under 15 mm. pressure, a small quantity of a viscid oil is obtained, which crystallises, and the distillation of which is not yet complete. The nitrile was purified by repeated distillation, and then analysed:

0.0985 gave 10.2 c.c. N_2 at 11° and 748 mm. $N = 12.1$.

$C_8H_{11}N$ requires $N = 11.6$ per cent.

Δ^1 -cycloHexeneacetonitrile is a colourless liquid, which distils at 17/90 mm., and has the pungent odour characteristic of nitriles; it combines with hydrobromic acid, with formation of an oily hydrobromide.

Hydrolysis.—The conversion of the nitrile into the corresponding acid appears to take place best under the following conditions.

The nitrile (20 grams) is digested with twice its volume of alcohol, containing 20 per cent. of sulphuric acid, for thirty-six hours, the mixture is diluted with water, extracted with ether, the ethereal solution washed well, dried, evaporated, and the residue distilled under 15 mm. pressure. The distillate, which consists of ethyl Δ^1 -cyclohexeneacetate and some unchanged nitrile, is digested with alcoholic potassium hydroxide for twenty minutes, diluted with water, and the unchanged nitrile and amide removed by extraction with ether.* The aqueous solution was evaporated until free from alcohol, cooled with ice, and filtered, when a viscid, syrupy acid separated, which, especially if stirred with a glass rod, soon solidified. It was collected, washed with water, drained on porous porcelain, and distilled under diminished pressure:

This oil gradually deposited crystals of Δ^1 -cyclohexeneacetamide, which separated from aqueous alcohol in glistening leaflets and melted at 152° (compare Wallach, *Ann.*, 1907, 353, 292).

0.1282 gave 0.3228 CO_2 and 0.0960 H_2O . $\text{C} = 68.5$; $\text{H} = 8.4$.

$\text{C}_8\text{H}_{12}\text{O}_2$ requires $\text{C} = 68.6$; $\text{H} = 8.6$ per cent.

Δ^1 -cyclohexeneacetic acid melts at about 38° , distils at 145° (17 mm.) and is identical with the acid of this constitution described by Wallach (*Annalen*, 1907, 353, 290), and which he obtained by an entirely different process. In order to be certain of the identity, the acid, prepared as described above, was left in contact with fuming aqueous hydrobromic acid, when it was converted into β -bromocyclohexylacetic acid, which, after crystallisation from light petroleum, melted at $89-91^\circ$:

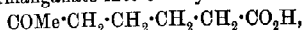
0.1512 gave 0.1270 AgBr . $\text{Br} = 35.7$.

$\text{C}_8\text{H}_{12}\text{O}_2\text{Br}$ requires $\text{Br} = 36.1$ per cent.

Wallach (*loc. cit.*) gives $89-90^\circ$ as the melting point of this bromo acid. We were also able to confirm Wallach's observation that Δ^1 -cyclohexeneacetic acid does not undergo isomeric change when boiled with strong alkalis, since, even after digesting for sixty hours with concentrated aqueous barium hydroxide, more than 75 per cent of the acid was recovered unchanged.

Oxidation of Ethyl Δ^1 -cyclohexeneacetate by Means of Ozone.

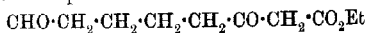
Wallach (*Annalen*, 1905, 343, 53; 1907, 353, 294; 1908, 356, 287) has shown that Δ^1 -cyclohexeneacetic acid is converted by oxidation with permanganate into δ -acetylvaleric acid,



a decomposition which clearly proves its constitution, but, as it was important to demonstrate the complete analogy between this acid and 1-methyl- Δ^3 -cyclohexene-4-acetic acid (p. 1948), we carried out a series of experiments on the oxidation of its ester by ozone.

For this purpose, the ester (5 grams) was dissolved in twice its volume of chloroform in a wide test-tube, a little water was added, and the ozone passed for several days and until it escaped freely.

The chloroform solution was diluted with ether, washed with sodium carbonate, dried, and the chloroform and ether removed by distillation under reduced pressure, when a neutral oil was obtained, which, in alcoholic solution, gave an intense blood-red coloration with ferric chloride, and there can be little doubt that this consisted essentially of the compound



(compare p. 1946). No attempt was made to purify and analyse this substance, and it was at once digested with dilute sulphuric acid and distilled in steam, when a volatile oil passed over, having the pungent odour characteristic of unsaturated ketones, and yielding a semicarbazone

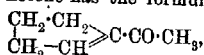
SYNTHESIS OF 1-METHYLCYCLOHEXYLIDENE-4-ACETIC ACID. 1961

which, after two recrystallisations from alcohol, melted with decomposition at 203—205°:

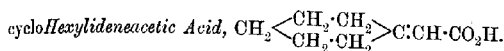
0.1439 gave 0.3021 CO₂ and 0.1073 H₂O. C=57.3; H=8.2.

C₈H₁₄ON₃ requires C=57.5; H=7.8 per cent.

It is explained in the introduction (p. 1946) that there can be little doubt that this volatile ketone has the formula



It is therefore Δ^1 -cyclopentene methyl ketone. It is most probably identical with the ketone which Wallach (*Annalen*, 1907, 353, 293) obtained by the oxidation of Δ^1 -cyclohexeneacetic acid with permanganate, and which also yielded a semicarbazone of melting point -204°. Wallach made the important observation that this compound yields glutaric acid when oxidised by chromic acid, a fact which strongly supports the above view of its constitution.



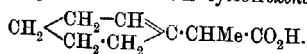
When the crude, semi-solid cyano-acid, obtained by the condensation of methyl sodiocyanoacetate and cyclohexanone under the conditions given on p. 1956, is distilled without further purification, and the nitrile obtained treated with sulphuric acid and alcohol and afterwards with alcoholic potassium hydroxide (p. 1959), a relatively large amount of pure hydrolysis, which is not the case when pure Δ^1 -cyclohexene-nitrile is employed. The unchanged nitrile was again subjected to the action of alcoholic sulphuric acid until it was completely nitrified, and the product yielded, on hydrolysis with alcoholic potash, a small quantity of an acid which melted at 89—90°:

0.1331 gave 0.3339 CO₂ and 0.1024 H₂O. C=68.5; H=8.5.

C₈H₁₂O₂ requires C=68.6; H=8.6 per cent.

There can be no doubt that this acid is cyclohexylideneacetic acid, is identical with the acid (m. p. 91°) which Wallach (*Annalen*, 1907, 3, 288) obtained from cyclohexanolacetic ester by elimination of water and subsequent hydrolysis.

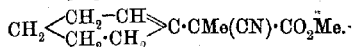
Δ^1 -cycloHexenepropionitrile and α - Δ^1 -cycloHexenepropionic Acid,



These substances are readily obtained from α -cyano- Δ^1 -cyclohexeneacetic ester by the following process.

The cyano-ester (30 grams) is added to sodium (3.5 grams), dissolved in methyl alcohol, and the intense yellow solution gradually mixed

with a considerable excess of methyl iodide, when rise of temperature takes place and the yellow colour disappears. After heating on the water-bath for forty-five minutes, the product is diluted with water, extracted with ether, the ethereal solution washed well, dried, evaporated, and the oil fractionated, when a considerable quantity distils at 140—142° (10 mm.) and consists of nearly pure methyl α -cyano- α - Δ^1 -cyclohexenepropionate,



0.1818 gave 11.9 c.c. N_2 at 13° and 762 mm. $\text{N} = 7.7$.

$\text{C}_{11}\text{H}_{16}\text{O}_2\text{N}$ requires $\text{N} = 7.2$ per cent.

This oil was digested with excess of alcoholic potassium hydroxide for ten minutes, water was then added, the alcohol removed by evaporation, the alkaline solution acidified, and repeatedly extracted with ether.

The ethereal solution was dried, evaporated, and the residue distilled under 90 mm. pressure, when carbon dioxide was eliminated and α - Δ^1 -cyclohexenepropionitrile was obtained as a colourless oil (12 grams), which, after repeated fractionation, distilled at about 150°/90 mm.:

0.1348 gave 0.3960 CO_2 and 0.1204 H_2O . $\text{C} = 80.1$; $\text{H} = 9.9$.

0.1727 „ 16.1 c.c. N_2 at 18° and 751 mm. $\text{N} = 10.5$.

0.1129 „ 10.0 c.c. N_2 at 17° „ 764 mm. $\text{N} = 10.3$.

$\text{C}_9\text{H}_{13}\text{N}$ requires $\text{C} = 80.0$; $\text{H} = 9.6$; $\text{N} = 10.4$ per cent.

On attempting to hydrolyse this nitrile, it was found that it exhibited remarkable stability towards the usual acid and alkaline hydrolytic agents, but, after boiling with alcoholic sulphuric acid for many hours, it was converted into ethyl α - Δ^1 -cyclohexenepropionate, and this, on hydrolysis with alcoholic potash, yielded the free acid. Prepared in this manner, α - Δ^1 -cyclohexenepropionic acid is a viscid oil, which distils at about 145°/18 mm.:

0.1379 gave 0.3495 CO_2 and 0.1069 H_2O . $\text{C} = 69.1$; $\text{H} = 8.6$.

$\text{C}_9\text{H}_{14}\text{O}_2$ requires $\text{C} = 70.1$; $\text{H} = 8.6$ per cent.

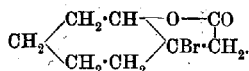
The silver salt was obtained, on the addition of silver nitrate to the slightly alkaline solution of the ammonium salt, as a curdy, white precipitate:

0.3862 gave 0.1596 Ag. $\text{Ag} = 41.3$.

$\text{C}_9\text{H}_{13}\text{O}_2\text{Ag}$ requires $\text{Ag} = 41.3$ per cent.

α - Δ^1 -cyclohexenepropionic acid has quite recently been prepared by Wallach (*Annalen*, 1908, 360, 44) from the product of the condensation of cyclohexanone with ethyl α -bromopropionate, and he describes it as an oil distilling at 143—150°/13 mm.

The Action of Bromine and Sodium Carbonate on Δ^1 -cyclohexeneacetic Acid. Formation of the Lactone of 2-Bromo-1-hydroxycyclohexyl-2-acetic Acid,



In this experiment, pure Δ^1 -cyclohexeneacetic acid (m. p. 38° ; 4.7 grams) was dissolved in dilute sodium carbonate (3.5 grams), and, after mixing with powdered ice, bromine (5.3 grams), dissolved in water, was gradually added. The cloudy liquid was extracted with ether, the ethereal solution washed well, dried, evaporated, and the syrup left over sulphuric acid in an evacuated desiccator, when it deposited a quantity of crystals, but much oil remained. The mass was left in contact with porous porcelain until quite dry, and then washed with light petroleum (b. p. $35\text{--}40^\circ$):

0.1585 gave 0.2530 CO_2 and 0.0710 H_2O . C = 43.5; H = 5.0.

$\text{C}_8\text{H}_{11}\text{O}_2\text{Br}$ requires C = 43.8; H = 5.0 per cent.

Purified in this manner, the lactone of 2-bromo-1-hydroxycyclohexyl-2-acetic acid is a colourless, crystalline substance, which melts at about 70° , and is insoluble in cold sodium carbonate, but dissolves readily in hot dilute potassium hydroxide. Some of the lactone was heated with water at $140\text{--}150^\circ$ for three hours, when a ketone was obtained, which, after distillation in steam, was converted into the semicarbazone in the usual manner. This separated from methyl alcohol as a colourless, crystalline mass, and melted at 192° :

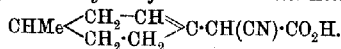
0.1141 gave 0.2365 CO_2 and 0.0929 H_2O . C = 56.6; H = 9.0.

0.1074 „ 24 c.c. N_2 at 24° and 760 mm. N = 25.0.

$\text{C}_8\text{H}_{13}\text{ON}_3$ requires C = 56.8; H = 8.9; N = 24.8 per cent.

That this substance was the semicarbazone of 1-methylcyclohexan-2-one, $\text{CH}_2 \begin{array}{c} \diagup \text{CH}_2 \cdot \text{CO} \diagdown \\ \diagdown \text{CH}_2 \cdot \text{CH}_2 \diagup \end{array} \text{CHMe}$, was proved by mixing it with a specimen of the semicarbazone prepared from 1-methylcyclohexan-2-one from *o*-cresol, when there was no alteration in melting point.

The Condensation of 1-Methylcyclohexan-4-one with Ethyl Cyanoacetate. α -Cyano-1-methyl- Δ^2 -cyclohexene-4-acetic Acid,



The condensation of 1-methylcyclohexan-4-one with ethyl sodiocyanoacetate was carried out under the following conditions.

Sodium (11.5 grams), dissolved in alcohol, was mixed with ethyl cyanoacetate (56 grams) and methylcyclohexanone (56 grams), when

the white sodium derivative soon dissolved, and a yellow solution was obtained. The whole was heated for half an hour on the water-bath, the bulky, yellow sodium derivative which separated decomposed by water and dilute hydrochloric acid, extracted with ether, the ethereal solution washed with water and dilute sodium carbonate, dried, and evaporated. On fractionating the residual oil, a small quantity of methylcyclohexanone was recovered, and ethyl α -cyano-1-methyl- Δ^3 -cyclohexene-4-acetate then distilled at 175°/20 mm. or 167°/14 mm. as a viscid, colourless oil:

0.1745 gave 0.4460 CO₂ and 0.1300 H₂O. C = 69.7; H = 8.3.

0.2477 „ 14 c.c. N₂ at 20° and 775 mm. N = 6.6.

C₁₂H₁₇O₂N requires C = 69.6; H = 8.2; N = 6.8 per cent.

The sodium carbonate extract of the product of condensation (see above) yielded, on acidifying with hydrochloric acid, a considerable quantity of a viscid syrup, which was extracted with ether.

After the ethereal solution had been dried and evaporated, the pale yellow residue was poured into a basin, when it rapidly crystallised, and, in contact with porous porcelain, the oily impurity was rapidly absorbed, leaving a colourless, crystalline mass, which was purified by solution in benzene and addition of light petroleum until a cloudiness appeared. On vigorously stirring, the acid separated in prisms:

0.1693 gave 12 c.c. N₂ at 19° and 760 mm. N = 8.1.

C₁₀H₁₃O₂N requires N = 7.8 per cent.

α -Cyano-1-methyl- Δ^3 -cyclohexene-4-acetic acid melts at 116°, and is sparingly soluble in water or light petroleum, but readily so in ether, alcohol, or benzene. It dissolves in aqueous potassium hydroxide (30 per cent.), yielding a clear solution, which soon clouds, owing to separation of 1-methylcyclohexan-4-one.

When the condensation of ethyl sodiocyanoacetate with methylcyclohexanone is carried out under the above conditions, the principal product obtained is the cyano-acid just described. If, however, the methylcyclohexanone is added to the ethyl sodiocyanoacetate suspended in hot alcohol, the sodium derivative dissolves immediately, and on cooling rapidly, extracting at once as before, and treating with sodium carbonate, the principal product is ethyl α -cyano-1-methyl- Δ^3 -cyclohexene-4-acetate. The sodium carbonate extract yields, on acidifying and extracting with ether, a solid mass, which, by crystallisation from benzene, may be separated into two substances, melting at 105° and 186° respectively. The substance of melting point 105° crystallised in colourless needles, and possibly consisted of α -cyano-1-methyl- Δ^3 -cyclohexene-4-acetic acid, crystallising with $\frac{1}{2}$ H₂O:

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0.1160 gave 0.2730 CO₂ and 0.0780 H₂O. C=64.2; H=7.5.

0.1486 „ 10 c.c. N₂ at 26° and 764 mm. N=7.5.

C₁₀H₁₃O₂N, 1/2 H₂O requires C=63.8; H=7.4; N=7.4 per cent.

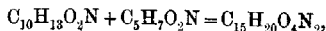
The substance of melting point 186° separated from benzene in feathery needles:

0.1300 gave 0.2930 CO₂ and 0.0830 H₂O. C=61.4; H=7.0.

0.1100 „ 9.4 c.c. N₂ at 26° and 764 mm. N=9.5.

C₁₅H₂₀O₄N₂ requires C=61.6; H=6.8; N=9.6 per cent.

The formula of this substance corresponds with that obtained by adding the formulae of α-cyano-1-methyl-Δ³-cyclohexene-4-acetic acid and cyanoacetic ester:



and there can be no doubt that it is a definite compound, and obviously somewhat similarly constituted to the substance obtained under similar conditions from cyclohexanone (p. 1957). When it is boiled with sodium carbonate, it is decomposed, with formation of α-cyano-1-methyl-Δ³-cyclohexene-4-acetic acid.

Condensation of 1-Methylcyclohexan-4-one with Ethyl Cyanoacetate and with Cyanoacetic Acid in the Presence of Piperidine.

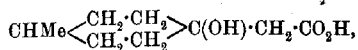
When ethyl cyanoacetate (28 grams) is mixed with methylcyclohexanone (28 grams) and a few drops of piperidine, the solution gradually clouds, owing to the separation of water. After heating on a water-bath for half an hour, the product was dissolved in ether, washed with dilute hydrochloric acid, the ethereal solution dried and vaporated, and the residue fractionated under diminished pressure, when an almost quantitative yield of ethyl α-cyano-1-methyl-Δ³-cyclohexene-4-acetate was obtained, boiling at 165—168°/14 mm.:

0.2066 gave 0.5274 CO₂ and 0.1560 H₂O. C=69.6; H=8.3.

C₁₃H₁₇O₂N requires C=69.6; H=8.2 per cent.

This ester is readily hydrolysed by boiling in alcoholic solution with slight excess of sodium methoxide, diluting with water, and evaporating until free from alcohol. After removing any unchanged ester by extraction with ether, the alkaline solution is acidified, and extracted in the usual way, when a colourless, crystalline mass of α-cyano-1-methyl-Δ³-cyclohexene-4-acetic acid is obtained, which, after crystallisation from benzene and light petroleum, melts at 116° (compare 1964). It was subsequently found that this acid may be obtained directly from methylcyclohexanone and cyanoacetic acid, by a simpler cooling way. Methylcyclohexanone (6 grams) is mixed with 1.5 g. of pure cyanoacetic acid (5 grams), piperidine (4.5 grams) is added, and the whole allowed to remain overnight and then heated, in which

the ester was digested with strong aqueous barium hydroxide containing a little potassium hydroxide, the same acid of melting point 41° was obtained, and, in addition, a considerable quantity of another acid melting at 138° . This latter acid is 4-hydroxy-1-methylcyclohexyl-4-acetic acid,



since, when mixed with a specimen of this acid which had been obtained by the condensation of methylcyclohexanone with ethyl bromoacetate (Marckwald and Meth, *Ber.*, 1906, 39, 1173), the mixture melted at 139 – 140° . Obviously therefore, under the conditions employed in this experiment, addition of water to the double linking in methyl- Δ^3 -cyclohexene-4-acetic acid had taken place to a considerable extent.

Ethyl 1-Methyl- Δ^3 -cyclohexene-4-acetate.—In order to prepare this ester in a pure state, the pure acid was digested with 10 per cent. alcoholic sulphuric acid for six hours, the product diluted with water, and extracted with ether. The ethereal solution was well washed with dilute sodium carbonate, dried, evaporated, and the residual ester fractionated, when it distilled constantly at $111^{\circ}/14$ mm. :

0.1290 gave 0.3420 CO_2 and 0.1136 H_2O . C = 72.3 ; H = 9.8.

$\text{C}_{11}\text{H}_{18}\text{O}_2$ requires C = 72.5 ; H = 9.9 per cent.

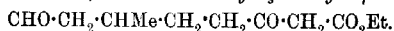
Oxidation of Ethyl 1-Methyl- Δ^3 -cyclohexene-4-acetate by Means of Ozone.

In carrying out this experiment, the pure ester was dissolved in chloroform, covered with water, and ozonised oxygen passed for several hours and until it escaped freely. The product was mixed with ether, the ethereal solution extracted with sodium hydrogen carbonate, dried, and evaporated, when a colourless oil remained which possessed the pungent odour of an aldehyde, and distilled, with the exception of a small quantity of resin, at 150 – $160^{\circ}/20$ mm. :

0.1280 gave 0.2840 CO_2 and 0.0976 H_2O . C = 60.5 ; H = 8.4.

$\text{C}_{11}\text{H}_{18}\text{O}_4$ requires C = 61.7 ; H = 8.4 per cent.

It is explained in the introduction (p. 1949) that this oily ester, which, in alcoholic solution, gives on the addition of ferric chloride a deep purple coloration, is doubtless *ethyl ξ -aldehydo- β -keto-isooctate*,



In order to hydrolyse this ester, it was mixed with dilute sulphuric acid and submitted to distillation in steam, when a mobile oil volatilised, which had an odour resembling that of the cyclic unsaturated ketones. The oil was extracted with ether, and, although only small in amount, it was distilled and found to consist essentially of a substance distilling at about 110 – $115^{\circ}/100$ mm. Small quantities of higher

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fractions were also obtained, and, as these yielded the same results on analysis as the lower boiling substance, they apparently consisted of polymerides of this (compare Wallach, *Annalen*, 1907, 353, 294). The fraction 110--115°/100 mm. yielded, on analysis, the following approximate figures, the difference being due to a trace of moisture, which, owing to the small amount of material at our disposal, we were unable to remove completely:

0.1220 gave 0.3360 CO₂ and 0.1110 H₂O. C = 75.2; H = 10.1.

C₈H₁₂O requires C = 77.4; H = 9.7 per cent.

That this is the formula for the ketone was proved by the preparation of the *semicarbazone*, which separates gradually when the ketone is shaken with strong aqueous semicarbazide hydrochloride and sodium acetate, and which crystallises from dilute methyl alcohol in short needles melting at 207--209°:

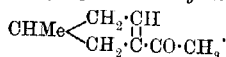
0.1096 gave 0.2400 CO₂ and 0.0860 H₂O. C = 59.7; H = 8.5.

0.0716 „ 0.1570 CO₂ „ 0.0550 H₂O. C = 59.8; H = 8.5.

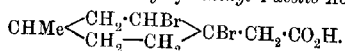
0.0790 „ 16 c.c. N₂ at 20° and 758 mm. N = 23.0

C₉H₁₂O₃N requires C = 59.7; H = 8.3; N = 23.2 per cent.

It is explained in the introduction (p. 1949) that the ketone C₈H₁₂O is doubtless 1-methyl-Δ³-4-cyclopentene methyl ketone,



3:4-Dibromo-1-methylcyclohexyl-4-acetic Acid,



1-Methyl-Δ³-cyclohexene-4-acetic acid readily absorbs bromine when the halogen is added to its solution in glacial acetic acid at 0°, and the addition is complete when the liquid has acquired a permanent yellow colour. On diluting with water, a viscid oil separates, which gradually crystallises, especially if vigorously stirred, and, after remaining in contact with porous porcelain until free from oil and then crystallising from formic acid, the dibromide is readily obtained pure:

0.2050 gave 0.2440 AgBr. Br = 50.6.

C₉H₁₄O₂Br₂ requires Br = 50.9 per cent.

3:4-Dibromo-1-methylcyclohexyl-4-acetic acid melts at 104°, and has already been prepared by Wallach (*Annalen*, 1907, 353, 312), who obtained it by the addition of bromine to the solution of the unsaturated acid in light petroleum, and states that it melts at 97--99°.

When this dibromo-acid is ground to a paste with water and shaken with sodium carbonate, it dissolves, and, in a short time, the solution

clouds and a heavy oil separates. This oil appears to be a mixture, and its investigation is not yet complete.

We have, however, found that, when heated with water at 140—150° (compare p. 1972), it is converted into 1:4-dimethylcyclohexan-2-one.

The ketone was extracted with ether, the ethereal solution dried, evaporated, and the oily residue fractionated, when a considerable quantity distilled at 174—177° and consisted of nearly pure 1:4-dimethylcyclohexan-2-one:

0.1674 gave 0.4640 CO₂ and 0.1740 H₂O. C = 75.6; H = 11.5.

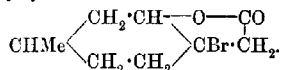
C₈H₁₄O requires C = 76.2; H = 11.1 per cent.

When this ketone was shaken with excess of semicarbazide hydrochloride and sodium acetate, it was gradually converted into the solid semicarbazone, which, after crystallisation from methyl alcohol, melted at 167°, and was identical with the semicarbazone of 1:4-dimethylcyclohexan-2-one:

0.0923 gave 0.1985 CO₂ and 0.0810 H₂O. C = 58.7; H = 9.7.

C₉H₁₇ON₃ requires C = 59.0; H = 9.3.

The Action of Bromine and Sodium Carbonate on 1-Methyl-Δ³-cyclohexene-4-acetic Acid. Formation of the Lactone of 4-Bromo-3-hydroxy-1-methylcyclohexyl-4-acetic Acid,



In this experiment, pure 1-methylcyclohexene-4-acetic acid (5 grams) was dissolved in dilute sodium carbonate (3.5 grams), and then bromine (5.3 grams), dissolved in water, gradually added, care being taken that the temperature did not rise above 10°.

The addition of the bromine water caused an immediate cloudiness, and, on standing, a heavy oil separated, which gradually crystallised.

The whole was extracted with ether, the ethereal solution washed well, dried, and evaporated, when a pale yellow syrup (7.5 grams) remained, which soon crystallised. The mass was left in contact with porous porcelain until quite free from oil, and then crystallised from light petroleum:

0.1756 gave 0.3000 CO₂ and 0.0920 H₂O. C = 46.5; H = 5.8.

0.1880 „ 0.1530 AgBr. Br = 34.5.

C₉H₁₃O₂Br requires C = 46.4; H = 5.6; Br = 34.3 per cent.

The lactone of 4-bromo-3-hydroxy-1-methylcyclohexyl-4-acetic acid melts at 102—103°, and, when heated at 125—130°, decomposes with elimination of carbon dioxide and hydrogen bromide. It is readily soluble in ether, alcohol, benzene, or chloroform, but sparingly so in cold light petroleum, and separates as a woolly mass of slender needles.

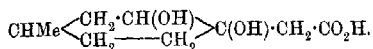
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when the hot solution in light petroleum (b. p. 70–80°) is allowed to cool slowly. The lactone is very slowly attacked by boiling water, and is insoluble in warm dilute sodium carbonate, but it dissolves gradually in hot dilute potassium hydroxide. In order to investigate its behaviour with alkali, a considerable quantity of the lactone was dissolved in dilute potassium hydroxide at 80°, and, after standing for two days, excess of potassium hydroxide was added and the whole heated on the water-bath for two hours. The product was acidified, extracted, and the ethereal solution dried and evaporated, when a viscid, yellow syrup remained, which was free from bromine, but showed no signs of crystallising. It was dissolved in boiling water, made distinctly alkaline by the addition of barium hydroxide, and the excess removed by passing carbon dioxide. After filtering, the almost colourless filtrate was acidified and extracted with ether, the ethereal solution was then very carefully dried, evaporated, and the syrup left over sulphuric acid in an evacuated desiccator until quite free from ether. The analysis shows that the syrup consists of the free acid and not of the lactone :

0.1452 gave 0.3150 CO₂ and 0.1140 H₂O. C = 57.2 ; H = 8.7.

C₉H₁₆O₄ requires C = 57.4 ; H = 8.5 per cent.

The basicity of this acid was determined by titration with decinormal sodium hydroxide, when 0.306 neutralised 0.0652 NaOH, whereas this amount of a monobasic acid, C₉H₁₆O₄, requires 0.0651 NaOH for neutralisation. There can be little doubt that this acid is 3 : 4-dihydroxy-1-methylcyclohexyl-4-acetic acid,



This acid dissolves readily in hot water, but strong solutions cloud on cooling.

The slightly alkaline solution of the ammonium salt gives no precipitate with barium or calcium chlorides. Copper sulphate gives a pale blue, amorphous precipitate, and lead acetate a caseous precipitate. If silver nitrate is added to a not too dilute solution of the ammonium salt, a gelatinous silver salt separates, which dissolves in hot water and crystallises, on cooling, in microscopic balls of needles. When the syrupy hydroxy-acid is heated under 13 mm. pressure, water is eliminated, and a viscid oil distils remarkably constantly at 186–188°/13 mm., which is sparingly soluble in cold water and in dilute sodium carbonate, but dissolves readily on warming. The analysis gave the following results :

0.1590 gave 0.3670 CO₂ and 0.1185 H₂O. C = 63.0 ; H = 8.2.

C₉H₁₄O₃ requires C = 63.5 ; H = 8.2.

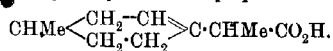
This substance is therefore the lactone of 3:4-dihydroxy-1-methylcyclohexyl-4-acetic acid.

In investigating the action of water on the lactone, $C_9H_{13}O_2Br$, it was heated, in quantities of one gram, with water (7 c.c.) in a sealed tube at 140—150° for three hours, when a volatile ketone was formed which, after extraction with ether, distilled at 170—180°. It was well shaken with a strong solution of semicarbazide hydrochloride and sodium acetate for several days, the crystalline mass was then collected, and recrystallised from methyl alcohol, from which the pure semicarbazone separated in colourless plates melting at 167°.

The ketone obtained in this way proved to be identical with that described on p. 1970, where analyses both of the ketone and its semicarbazone are given. As a series of experiments seemed to indicate that the above ketone was identical with 1:4-dimethylcyclohexan-2-one, recently described by Sabatier and Mailhe (*Compt. rend.*, 1906, 142, 553), we prepared some of the ketone, for the sake of comparison, by the method recommended by these investigators.

Pure *p*-xyleneol was slowly distilled over reduced nickel in a stream of hydrogen, and, after it had been twice subjected to this treatment, the 1:4-dimethylcyclohexan-2-ol obtained was oxidised by chromic acid and the resulting 1:4-dimethylcyclohexan-2-one distilled in a current of steam.* The ketone was extracted from the distillate and fractionated, when almost the whole quantity distilled at 176°, and yielded a semicarbazone, which, when first prepared, melted at 155°, but, after repeated crystallisation from methyl alcohol, the melting point rose to 167°. That 1:4-dimethylcyclohexan-2-one is identical with the ketone produced by the action of water on the lactone, $C_9H_{13}O_2Br$, in the manner just described, was proved by careful comparison, and especially by the fact that the semicarbazones from both specimens, not only melted at 167°, but also that, when mixed, there was no alteration in the melting point.

*α-1-Methyl-Δ³-cyclohexene-4-propionic Acid,**



In order, in the first place, to prepare methyl *α*-cyano-1-methyl-Δ³-cyclohexene-4-propionate, sodium (2·3 grams) was dissolved in methyl alcohol, mixed with ethyl *α*-cyano-1-methyl-Δ³-cyclohexene-4-acetate (21 grams),† and then methyl iodide (20 grams) added, when

* We are indebted to Mr. H. D. Gardner for carrying out this preparation.

† Two condensations were made in one of which the cyano-ester had been prepared by the sodium ethoxide condensation (p. 1963), and in the other with the aid of piperidine (p. 1965), and the results in both cases were identical.

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considerable rise of temperature took place. After one hour, the product was heated for half an hour on the water-bath, mixed with water, and the oil extracted with ether.

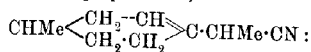
The ethereal solution was washed well, dried, evaporated, and the residue distilled under diminished pressure :

0.1608 gave 9.6 c.c. N_2 at 19° and 756 mm. $N = 6.8$.

0.1406 „ 8.5 c.c. N_2 „ 18° „ 760 mm. $N = 6.9$.

$C_{12}H_{17}O_2N$ requires $N = 6.8$ per cent.

Methyl α -1-methyl- Δ^3 -cyclohexene-4-propionate is a colourless oil, which distils at $158-160^\circ/20$ mm. When the pure cyano-ester (17 grams) was digested on the water-bath with potassium hydroxide (9 grams) for three hours, and then water added, it was found that a portion only of the oil had passed into solution. The product was acidified, extracted with ether, the ethereal solution well washed with dilute sodium carbonate, dried, and evaporated, when a considerable quantity of oil remained, which distilled at $162^\circ/100$ mm., and consisted of *1-methyl- Δ^3 -cyclohexene-4-propionitrile*,

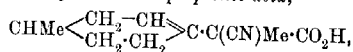


0.1480 gave 12.4 c.c. N_2 at 26° and 752 mm. $N = 9.5$.

0.0965 „ 8.0 c.c. N_2 „ 18° „ 765 mm. $N = 9.6$.

$C_{10}H_{15}N$ requires $N = 9.4$ per cent.

The sodium carbonate solution was acidified and extracted with ether, when a viscid, syrupy acid was obtained, which evidently contained *cyano α -1-methyl- Δ^3 -cyclohexene-4-propionic acid*,*



which, when distilled under reduced pressure, it was decomposed with elimination of carbon dioxide and formation of *α -1-methyl- Δ^3 -cyclohexene-4-propionitrile*. A quantity of the nitrile prepared in this way was again found to distil at $162^\circ/100$ mm. :

0.2130 gave 17.5 c.c. N_2 at 19° and 760 mm. $N = 9.5$.

$C_{10}H_{15}N$ requires $N = 9.4$ per cent.

This nitrile is hydrolysed with considerable difficulty, and even after boiling in a reflux apparatus with 20 per cent. alcoholic sulphuric acid for thirty-six hours, it was only partly converted into ethyl *α -1-methyl- Δ^3 -cyclohexene-4-propionate*. The product was mixed with ether and water, the ethereal solution separated, washed well, evaporated, and the residual oil digested with excess of methyl-alcoholic potash for fifteen minutes. After diluting, acidifying, and extracting with ether,

* This acid is produced almost quantitatively when the cyano-ester is hydrolysed with potassium hydroxide in the cold.

the acid was separated from much unchanged nitrile by shaking the ethereal solution with sodium carbonate.

The aqueous solution was acidified, when an oil was precipitated which was again extracted with ether, and, after drying, the ether was evaporated and the residue distilled :

0.2427 gave 0.6280 CO_2 and 0.2035 H_2O . $\text{C} = 70.6$; $\text{H} = 9.3$.

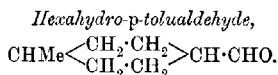
$\text{C}_{10}\text{H}_{16}\text{O}_2$ requires $\text{C} = 71.4$; $\text{H} = 9.5$ per cent.

α -1-Methyl- Δ^3 -cyclohexene-4-propionic acid, obtained in this way, distilled at $154\text{--}155^\circ/20$ mm. The silver salt was prepared by precipitating a slightly alkaline solution of the ammonium salt with silver nitrate :

0.5030 gave 0.1990 Ag. $\text{Ag} = 39.5$.

$\text{C}_{10}\text{H}_{15}\text{O}_2\text{Ag}$ requires $\text{Ag} = 39.3$ per cent.

During their experiments on the condensation of 1-methylcyclohexan-4-one with ethyl α -bromopropionate (*Annalen*, 1908, 360, 32), Wallach and Evans obtained an unsaturated acid distilling at $152\text{--}154^\circ/13$ mm., which partly solidified and melted at $103\text{--}104^\circ$, and this is possibly identical with our acid. The syrupy acid which we obtained showed no signs of crystallisation, even when well cooled and vigorously stirred with a glass rod, but this behaviour may be due to the syrup being a mixture of *cis*- and *trans*-isomerides.



The method employed in the preparation of this aldehyde was similar to that used with success in analogous cases by Tschitschibabin (*Ber.*, 1904, 37, 850). The 4-bromo-1-methylcyclohexane required for the synthesis was obtained by dissolving 1-methylcyclohexan-4-ol in fuming hydrobromic acid (saturated at 0°), and heating the solution in the water-bath, when it separated into two layers. Water was added, the heavy oil extracted with ether, the ethereal solution washed, dried, and evaporated, and the crude bromide distilled, when almost the whole quantity passed over at $130^\circ/200$ mm. 4-Bromo-1-methylcyclohexane (88 grams) was dissolved in dry ether (3 vols.), magnesium (12 grams) was then added, when, on warming, reaction set in, at first slowly, but then proceeded vigorously. The product was mixed with toluene (75 grams), the ether distilled off, and then orthoformic ester (74 grams) added, when a solid magnesium compound separated immediately (compare Bodroux, *Compt. rend.*, 1904, 138, 700).

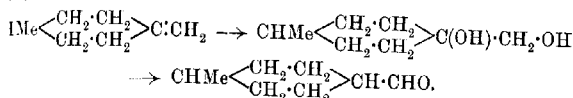
After heating on the water-bath for an hour and a-half, the product was decomposed by water and dilute hydrochloric acid, and extracted with ether in the usual manner. The ethereal solution was dried

evaporated, the toluene removed by heating on the water-bath under reduced pressure, and the residual acetal digested for three hours with dilute hydrochloric acid (5 per cent.). The oily layer was separated, and well shaken with concentrated sodium hydrogen sulphate, the solid additive compound was collected, dissolved in water, and decomposed by sodium carbonate. After extracting with ether, the ethereal solution was washed rapidly, dried, evaporated, and the residual aldehyde distilled :

0.2372 gave 0.6550 CO_2 and 0.2330 H_2O . $\text{C} = 75.4$; $\text{H} = 10.9$.

$\text{C}_8\text{H}_{14}\text{O}$ requires $\text{C} = 76.2$; $\text{H} = 11.1$ per cent.

Heptahydro-p-tolualdehyde distils at about 180° , and has the pungent odor characteristic of aldehydes of this class ; it readily undergoes polymerization, doubtless owing to condensation. The semicarbazone, prepared in the usual manner, separated from dilute methyl alcohol as a colorless, crystalline mass, which melted at about 154° . It has been mentioned (p. 1955) that Wallach (*Annalen*, 1906, 347, 348) had only prepared this aldehyde from 1-methyl-4-methylenecyclohexane oxidation to the glycol and subsequent treatment with dilute sulphuric acid :



Wallach gives $154-156^\circ$ as the melting point of the semicarbazone.

We wish to state that much of the expense of this investigation has been covered by several grants from the Research Fund of the Chemical Society, for which we desire to express our thanks.

THE UNIVERSITY,
MANCHESTER.

CVI.—The Direct Union of Carbon and Hydrogen. *Synthesis of Methane.*

By WILLIAM ARTHUR BONE and HUBERT FRANK COWARD.

In the year 1897 one of us, in conjunction with Dr. D. S. Jerdan, published the results of an investigation (*Trans*, 71, 41), from which it was concluded that "at a temperature of 1200° , or thereabouts, carbon unites directly with hydrogen to form methane, no ethylene or other unsaturated hydrocarbon being formed at this

temperature." Four years later, the synthesis of methane from its elements at 1200° was confirmed by further experiments (Trans., 1901, **79**, 1042), which also proved that no other members of the paraffin series are produced under these conditions.*

The fact that methane can be directly synthesised at so low a temperature was denied by Berthelot in 1905 (*Compt. rend.*, 1905, **40**, 905; *Ann. Chim. Phys.*, 1905, [viii], **6**, 183), who supported his contention by the results of experiments in which a small quantity (0.018 to 0.0412 gram) of highly purified carbon was heated in hydrogen to 1325° for an hour in a sealed quartz tube of 3 to 4 c.c. capacity. Berthelot said: "Il résulte de ces expériences que le carbone, sous les trois formes de diamant, de graphite, de carbone amorphe bien purifié, ne s'unit pas à l'hydrogène à 1325° , sous la seule influence de la chaleur." And in summarising his many attempts to synthesise methane, he further remarked: "J'ai fait depuis 50 ans de nombreux essais, dans des conditions très diverses, pour combiner le carbone amorphe au rouge vif. Or, je n'ai jamais pu constater rigoureusement cette combinaison, toutes les fois que le carbone a été absolument pur, d'hydrogène et d'azote combinés, ainsi que d'alcalis et de fer." The value of Berthelot's later experiments was, however, impaired by the fact that his quartz tubes were admittedly pervious to gases at high temperatures; he acknowledged that between 27 and 40 per cent. of the hydrogen escaped out of the tubes during the hour over which each experiment extended, and from the context it is obvious that there was also a considerable leakage of air in the opposite direction.

Any doubts which may thus have been raised regarding the direct synthesis of methane from its elements at 1100° to 1300° were dispelled by the appearance of a paper by Pring and Hutton in 1906 (Trans., **89**, 1591), although a careful repetition of Bone and Jerdan's experiments by the present authors, soon after the appearance of Berthelot's paper in 1905, confirmed their accuracy in all essential particulars (see p. 1981). The inconclusive character of Pring and Hutton's work, however, caused us to postpone any reply until we were in a position to bring forward such new evidence as would place the issue beyond doubt.

Pring and Hutton's experiments consisted in heating electrically rods of purified carbon in an atmosphere of hydrogen to various temperatures between 1000° and 2800° , the apparatus being so constructed that the heated rod was far removed from the glass or metal walls of the containing vessel. The results left the main

* Attention had been previously drawn to the probability of the direct synthesis of methane from its elements by Bone and Cain (*Proc.*, 1894, **10**, 180).

are quite undecided, for, whilst small quantities of methane are invariably obtained (thus, for example, 0.20 to 0.25 per cent. at 1350°, and 0.40 to 0.43 per cent. at 1700°, for "carbons purified with chlorine," tables III and IV., p. 1599), the authors throw doubt on its synthetic formation by stating (p. 1598) that "throughout the whole of the work, the greater the precautions which were taken to improve the quality of the carbon the lower was the percentage of methane formed." But no evidence was advanced to support the assumption that the observed falling off in the rate of methane formation synchronised with any material increase in the purity of the carbon; on the contrary, in almost the next paragraph (p. 1598), the authors apparently rejected the idea that the methane had arisen from the decomposition of hydrocarbon impurity in the carbon rods, on account of their failure to detect more than 0.1 per cent. of methane in the gases evolved when an entirely new rod was heated in a vacuum (p. 1599). The alternative theory, that the observed facts might be due to a progressive falling off in the activity of the carbon in consequence of physical changes in the surface layer induced by prolonged exposure to such high temperatures, was apparently overlooked. Finally, it may be pointed out that the authors' claim of having synthesised acetylene at 1800° (p. 1600) is illogical, unless they are also prepared to admit the direct formation of the much larger quantities of methane at the same temperature. The case for acetylene rests on no stronger evidence than that for methane, and the two must obviously stand or fall together.

Not more than a year ago, Mayer and Altmayer published the results of some experiments on the reversible reaction $2\text{H}_2 \rightleftharpoons \text{CH}_4$ at 475° to 625° in presence of nickel or cobalt as catalyst. From their results, together with certain assumptions regarding the specific heat of the reacting substances and the rate of formation of methane at the experimental temperature, they deduced the following mathematical expression for the state of equilibrium finally attained by the system at any temperature on the absolute scale:

$$CT = -18507 + 5.9934 T \log T + 0.002936 T^2 + RT \log \frac{p_{\text{CH}_4}}{p_{\text{H}_2}^2},$$

where p_{CH_4} and p_{H_2} are the partial pressures of the methane and hydrogen respectively, and C is a constant = 21.1, deduced from their experimental results.

They then proceeded by extrapolation to calculate the percentages of methane in a system in equilibrium at atmospheric pressure at various temperatures between 250° and 850°—that is to say, a range of temperature four times as great as that actually covered by their

experiments--and finding that at 850° the percentage of methane would be 1.59 only, they did not hesitate to deny the possibility of synthesising methane from its elements at 1200° , and to suggest that the results obtained by Bone and Jerdan were due to impurities in the carbon which had been employed.* Fortunately, we are in a position to refute this aspersion by giving the results of analyses of the particular carbon in question.

It seemed to us that the most effective way of replying to the various critics would be to convert a small quantity of highly purified carbon quantitatively, or nearly so, into methane by heating it in a stream of pure dry hydrogen at 1100° to 1200° in a form of apparatus similar to that used by Bone and Jerdan in 1891. The task proved by no means an easy one, partly on account of the length of time required, which imposed considerable physical strain on the operators, but mainly because of the marked reducing action of the methane on the glaze of the porcelain tube in which the carbon was heated. We finally succeeded, however, in obtaining 73 per cent. of the theoretical yield of methane from about 0.1 gram of highly purified carbon in two different experiments, each of which extended over thirty-six hours without a break. Such a result obviously precludes the supposition that the methane arose from any possible traces of impurity in the carbon used, and, since throughout the final experiment the hydrogen had been perfectly dried by passage through a long spiral immersed in a bath of liquid air, the direct synthesis of methane, as originally carried out by Bone and Jerdan, would appear to be established beyond all doubt.

In our recent paper on the "Thermal Decomposition of Hydrocarbons" (this vol., p. 1197), proof was given of the far greater stability of methane as compared with ethane, ethylene, and acetylene at temperatures up to 1200° , and of the marked tendency to form it by the direct hydrogenation of (presumably solid) residues as $\cdot\text{CH}_3$, $:\text{CH}_2$, and $:\text{CH}$ at temperatures above 800° . The new evidence, contained in the present paper, of the formation of methane by the direct hydrogenation of solid carbon itself now more emphasises the importance of this hydrocarbon as a high temperature product.

Attention is drawn to the fact that the question of the conditions of equilibrium in a system containing carbon, hydrogen, and methane at 1100° to 1200° is not considered in this paper. We hope to deal with this aspect of the subject in a future communication.

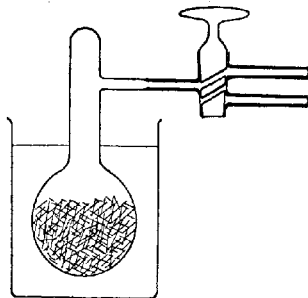
* In the concluding paragraph of their paper (*Ber.*, 1907, 40, 2144), referring to the work of Bone and Jerdan, they remark: "Nach den hier berichteten Ergebnissen kann dieses Methan nicht aus Kohlenstoff und Wasserstoff entstanden sein. Wahrscheinlich enthielt ihre Kohle Verunreinigungen, die Methan lieferten."

EXPERIMENTAL.

The Estimation of Small Quantities of Methane in a Gas mainly consisting of Hydrogen.

The accurate estimation of such small percentages of methane is formed when a stream of pure hydrogen is passed over carbon at 1100° to 1200° cannot be carried out in the usual way by determining the amount of carbon dioxide formed when the gas is exploded with excess of oxygen, unless the greater part of the diluting hydrogen is previously removed by absorption in contact with palladium. Hutton and Pring (*loc. cit.*, p. 1598), using a Sodeau apparatus, and without any such preliminary concentration, did indeed claim that their numbers for methane might be relied on within 0.04 per cent., despite the fact that oxides of nitrogen

FIG. 1.



were admittedly produced in their explosions to the extent of 0.1 per cent. We feel very doubtful whether, under these conditions, such a high degree of accuracy is at all possible.

Throughout the present investigation all analyses were carried out on the residual gas obtained by concentrating about a litre of the original gas to between 20 and 50 c.c. over palladium foil at 100° . The apparatus used for the concentration is shown in Fig. 1. A measured volume of the gas under examination was slowly admitted into the vacuous globe, *A* (capacity = 150 c.c.), which was packed with small pieces (31 grams) of thin palladium foil,* and was heated in a water-bath to 100° during the absorption of the hydrogen. Most of the hydrogen was quickly absorbed, after

* We are indebted to the kindness of Prof. H. B. Dixon for the loan of this palladium foil.

which the water-bath was removed and the residual gas withdrawn by means of an automatic Sprengel pump into a graduated tube over mercury. The volume of the "concentrate" having been determined, it was successively treated with solid potassium hydroxide, ammoniacal cuprous chloride solution, and dilute sulphuric acid to ensure the elimination of any traces of oxides of carbon, after which it was finally analysed by explosion with excess of oxygen in the usual manner. In this way, the accuracy of the estimation of the methane formed was increased something between twenty- and fifty-fold as compared with the usual method. The hydrogen used in each experiment was also similarly concentrated, and the residual gas analysed.

The method seemed open to one possible objection which, however, proved quite groundless. The gas obtained in our experiments always contained, besides methane, a small quantity (usually less than 0.2 per cent.) of carbon monoxide, derived by the reducing action of some of the methane on the walls of the porcelain tube in which the carbon was heated. The exit gases were invariably passed through a strong and freshly-prepared ammoniacal solution of cuprous chloride before concentration over the palladium, which reduced the carbon monoxide to certainly less than 0.05 per cent. But in order to make quite sure that this small quantity would not give rise to methane during the concentration, two blank experiments were performed, in each of which a litre of hydrogen containing as much as 2.0 per cent. of carbon monoxide was concentrated, in one case to 40 and in the other to 65 c.c., over the palladium foil. On subsequently analysing the residual gas, after the removal of carbon monoxide, the amount of methane found amounted to no more than between 0.01 and 0.02 per cent. on the original gas concentrated; in other words, any methane formed during the concentration did not exceed one-hundredth part of the carbon monoxide originally present. It may therefore be safely assumed that the presence of less than 0.05 per cent. of carbon monoxide in the gases concentrated in our experiments had no appreciable effect on the results for methane.

B. Analysis of the Carbon used by Bone and Jerdan and a Repetition of their Experiments.

The experiments recorded in this section of the paper were carried out in June 1905 immediately after the appearance of Berthelot's paper and before Pring and Hutton commenced their experiments.

(1) *Analysis of Bone and Jerdan's Carbon.*—We were fortunate in possessing a few grams of the sample of carbon actually used by

Bone and Jerdan in 1896-1901. It had been prepared by the ignition of cane-sugar, as described in their first paper (*loc. cit.*, p. 45), and had been carefully purified by being heated to redness in a current of dry chlorine for many hours, after which all traces of chlorine were removed by prolonged heating in hydrogen at the same temperature. Two analyses of the carbon were made by burning it in a stream of dry oxygen in a combustion tube packed with red-hot copper oxide, and weighing the water produced, as follows:

	Per cent.
(1) 1.00 gave 0.0045 H_2O .	H = 0.05
(2) 0.90 „ 0.0058 H_2O .	H = 0.07

Mean = 0.06

It is tolerably certain that at least part of this hydrogen was present in an "occluded" form (it is extremely difficult to remove the last traces of occluded hydrogen from carbon even in a vacuum at red heat), but even assuming it to be wholly present as hydrocarbon impurity, the amount of methane derivable therefrom would not exceed 3.3 c.c. per gram of the carbon.

(2) *Repurification of the Carbon.*—Between 2 and 3 grams of the carbon were subsequently heated to redness in a stream of dry chlorine for one hundred and seventeen hours, and afterwards in pure dry hydrogen to the same temperature for three days. All joints in the apparatus were of fused glass. Finally, the carbon was transferred to the porcelain tube of the apparatus, to be described hereafter, and further heated in pure dry hydrogen at 1100° for twenty-six hours before the experiments recorded in the next paragraph were commenced. This drastic treatment would certainly reduce any traces of hydrocarbon impurity (if, indeed, such were actually present) to vanishing proportions.

(3) *Repetition of Bone and Jerdan's Experiments.*—0.34 Gram of this repurified carbon was heated to 1020 – 1070° in a slow current of pure dry hydrogen in an apparatus in all respects similar to that used by Bone and Jerdan in 1897, except that the furnace now employed was of an improved type and allowed of a longer length of the combination of the two coaxial porcelain tubes being heated. A current of dry hydrogen was maintained through the annular space between the two porcelain tubes throughout each experiment. The issuing gas was collected, and subsequently concentrated over palladium before the final estimation of methane was made. The results of three successive experiments, together with a fourth blank experiment, were as follows:

(a) 900 c.c. of issuing gas collected in two and a-quarter hours. Temperature = 1020° .

Methane found in the concentrate = 0.65 per cent. on the original 900 c.c. collected.

\therefore Total methane in issuing gas = 5.85 c.c.

(b) 900 c.c. of issuing gas collected in two hours. Temperature = $1050-1075^{\circ}$.

Methane found in the concentrate = 0.69 per cent. on the original 900 c.c. collected.

\therefore Total methane in issuing gas = 6.2 c.c.

(c) 800 c.c. of issuing gas collected in two hours. Temperature = 1020° .

Methane found in the concentrate = 0.53 per cent. on the original 800 c.c. collected.

\therefore Total methane in issuing gas = 4.2 c.c.

(d) 2000 c.c. of the pure hydrogen employed were passed through the heated tube, which now contained no carbon, and 1100 c.c. of the issuing gas were subsequently concentrated to 15 c.c. over palladium. An analysis of the residue showed that the original gas contained 0.03 per cent. of methane and 0.16 per cent. of nitrogen only.

Summary.—Of the 16.25 c.c. of methane found in the issuing gases in the above experiments, not more than 0.9 c.c. could have been contained in the 2800 c.c. of hydrogen employed, whilst, at the utmost, not more than 1.1 c.c. could have been derived from any hydrocarbon impurity in the 0.34 gram of carbon used, even on the assumption that the drastic treatment described under (2) had been completely ineffectual. It follows, therefore, that at least 14.25 c.c. of methane must have been produced by the interaction of carbon and hydrogen. It may thus be fairly claimed that this series of experiments not only establishes the high degree of purity of the carbon used by Bone and Jerdan, but also confirms, in all essential particulars, their synthesis of methane.

C. Series of Three Experiments showing a Large Yield of Methane from a Small Quantity of Carbon at 1100° .

These experiments, which were carried out during the summer of last year, were designed to demonstrate the possibility of converting a small quantity of highly purified carbon very largely into methane by simply heating it to 1100° , or thereabouts, in a steady current of pure and well-dried hydrogen, within the limits of a twenty-four hours' experiment.

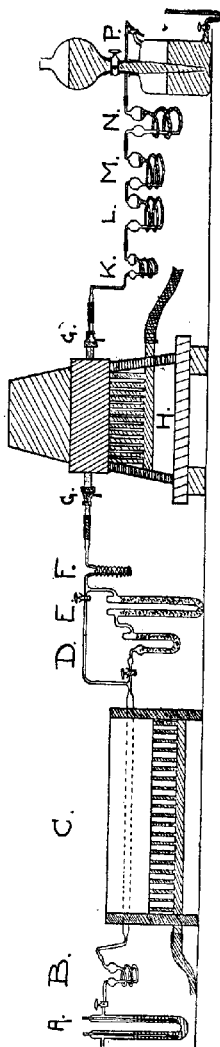
Preparation and Purification of the Carbon.—A new sample of sugar-charcoal was prepared in the manner described by Bone and

Jerdan. Its purification was effected in a stream of dry chlorine, first of all at 650° for two hundred and ninety-six hours in a Jena glass combustion tube, and afterwards at 1100° for twenty-five hours longer in the inner porcelain tube of the apparatus described below. Finally, it was heated to 1100° in a current of pure dry hydrogen for fifty hours until the last traces of chlorine were eliminated. Analysis showed that it contained only 0.08 per cent. of hydrogen, probably mainly in an "occluded" form. During the second stage of the purification in chlorine, the carbon had been contained in a platinum boat, in order to avoid its coming into contact with the inner surface of the hot porcelain tube, and it was subsequently found to have become associated with about 14 per cent. of finely-divided platinum, which, as was found later, had the effect of increasing its activity towards hydrogen.

Preparation of the Hydrogen.—The hydrogen used in these and all subsequent experiments was prepared by the electrolysis of a solution of recrystallised barium hydroxide in a U-shaped electrolytic cell which formed an integral part of the apparatus described below. By means of a constant current of about 2.25 amperes, a perfectly steady evolution of hydrogen at a rate of about 0.95 litre per hour could be maintained throughout the whole of each experiment.

The Apparatus.—The arrangement of the apparatus is shown in Fig. 2. A is the electrolytic cell supplying the pure hydrogen. The gas evolved passed successively through (1) the empty worm B, cooled externally; (2) the tube C of Jena hard glass, containing a

FIG. 2



long roll of copper gauze heated to redness in a combustion furnace; (3) the U tube *D*, containing ignited calcium chloride; (4) the U tube *E*, containing solid potassium hydroxide; (5) the glass spiral *F*, with nine turns, each 3.3 cm. in diameter, which in the final experiment was externally cooled with liquid air. The dry gas then entered the inner one (*G G*) of the arrangement of two coaxial porcelain tubes, similar to that used by Bone and Jerdan,* which contained the purified carbon resting in a platinum boat so as to be out of contact with the walls of the tube. The tubes were heated in a powerful gas furnace *H* to a temperature of 1100° to 1175°, a slow current of pure dry hydrogen being maintained through the annular space between the two tubes throughout the whole of each experiment. The gas issuing from the heated inner tube passed through (1) the worm *K*, containing 50 per cent. sulphuric acid; (2) the worms *L* and *M*, containing a strong and freshly prepared ammoniacal solution of cuprous chloride; (3) the

FIG. 3.



worm *N*, containing 50 per cent. sulphuric acid, and was finally collected in the graduated holder *P* over a mixture of equal volumes of glycerol and water.

All the joints in the apparatus, except those leading in and out of the worms *L* and *M*, the contents of which required changing between each experiment, were of fused glass. Those leading in and out of the combustion tube *C*, were composed of several kinds of glass of carefully graded hardness all fused up together.

The construction of the joints leading in and out of the inner porcelain tube containing the carbon is shown in Fig. 3. Each was composed of a closely-fitting glass tube carefully ground into the open end of the porcelain tube, the joint being secured externally by means of a short length of stout rubber tubing. In this way a perfectly tight joint was made without the gas coming into contact with rubber at all.

* The details of this arrangement were fully described in Bone and Jerdan's first paper (*loc. cit.*, p. 47), and were referred to again in our recent paper on "The Thermal Decomposition of Hydrocarbons" (this vol., p. 1203).

In the experiments to be described in the next section, the inner porcelain tube was furnished with a loosely-fitting quartz tube which extended throughout the whole of the heated portion; in the experiments at present under consideration this quartz lining was not inserted.

Experimental Plan.—The mode of procedure in these experiments was as follows:

About 0.1 gram of the purified carbon was weighed out into a platinum boat and introduced into the inner porcelain tube (all cold). The air in the apparatus having been completely displaced by the pure dry hydrogen, the furnace was lighted, and the temperature of the porcelain tubes raised to about 1150° as rapidly as possible. During this initial period of heating, the current of hydrogen was suspended. About fifteen minutes after the inner tube had attained the maximum experimental temperature, the current of hydrogen was re-started, and the collection of the issuing gas in the holder *P* was begun, a litre being collected during the next sixty-five to seventy minutes. The experiment was continued usually about twenty-five hours, the stream of hydrogen being maintained at a steady rate of about 0.95 litre per hour throughout. Litre samples of the issuing gas were collected at selected intervals, and after concentration over palladium foil to a known volume, the methane in the concentrate was determined. In this way, the rates of formation of methane at different periods during each experiment were accurately determined, and, knowing the total volume of hydrogen passed through the apparatus, an estimation could be made of the total methane produced. The results of each experiment may be summarised in tabular form as follows.

Experiment I.

Preliminary Blank Experiment to Test the Purity of the Hydrogen.
—Hydrogen passed through the apparatus with the porcelain tubes heated, but containing no carbon. One litre of gas collected in sixty-two minutes, and was subsequently concentrated over palladium foil to 22 c.c. Methane in the residual gas was equivalent to 0.006 per cent. on the original litre collected.

Actual Experiment.—Total duration=twenty-six hours. Temperature=1175°. Weight of carbon taken=0.086 gram. Total hydrogen used=23 litres.

Sample.	Hour from beginning.	1 litre collected, in minutes.	1 litre concentrated to c.c.	Percentages in the litre of gas collected.*		
				CH ₄ .	CO.	N ₂ .
I	0—1	67	30	0.68	0.012	0.10
II	3½—4½	66	23	0.58	0.014	0.09
III	10—11½	73	33	0.52	0.02	0.04
IV	24—25	65	25	0.20	0.04	0.10

Summary.—Estimate of the total amount of methane in the issuing gases—96 c.c. at 15° and 760 mm. Of this not more than 1.4 c.c. would be originally present in the 23 litres of hydrogen, and not more than 0.4 c.c. could possibly have been derived from impurity in the carbon. Therefore 92.2 c.c. must have been formed by the direct action of the hydrogen on the carbon. Now the total quantity of methane theoretically obtainable from 0.086 gram carbon=170 c.c. at 15° and 760 mm., so that about 54 per cent. of the carbon was converted into methane during the experiment, and at the end of twenty-five hours appreciable quantities of methane were still being produced. Some of the carbon was left over at the end of the experiment, and part of the methane formed had been lost by oxidation in contact with the inner surface of the heated porcelain tube.

Experiment II.

Total duration=twenty-six hours. Temperature=1100—1150°. Weight of carbon taken=0.086 gram. Total hydrogen used=26 litres.

Sample.	Hour from beginning.	1 litre collected, in minutes.	1 litre concentrated to c.c.	Percentages in the litre of gas collected.		
				CH ₄ .	CO.	N ₂ .
I	0—1	65	23.5	0.69	0.009	0.18
II	3—4	66	31.0	0.59	nil	0.15
III	9—10	73	24.5	0.42	0.05	0.02
IV	20—21	65	61.0	0.27	0.09	1.15
V	25—26	65	70.0	0.06	0.07	1.86

* The carbon monoxide in the gases issuing from the tube in this and the two following experiments would average about 0.2 to 0.25 per cent. ; it was, however, nearly all absorbed by the cuprous chloride solution in the worms *L* and *M* (Fig. 2) before the litre samples were collected.

Summary.—Estimate of the total amount of methane in the issuing gases = 95 c.c., of which not more than 1.6 c.c. would be originally present in the hydrogen used, and not more than 0.4 c.c. could have been derived from any impurity in the carbon. Therefore 93 c.c. must have been formed by the action of the hydrogen on the carbon.

It is evident that towards the end of the experiment, after about the twentieth hour, small quantities of air were leaking into the apparatus, probably due to incipient porosity in the porcelain tube, which was showing signs of brittleness. The inner surface of the tube had been much reduced by the methane.

Experiment III.

Before this experiment, the following "blanks" were performed:

(1) Pure dry hydrogen was passed through the apparatus, the porcelain tubes being heated as in an actual experiment, but no carbon was introduced. One litre of the issuing gas was collected and concentrated to 34 c.c. The methane found in the residual gas was equivalent to 0.01 per cent. on the original hydrogen collected.

(2) A mixture of 98 per cent. hydrogen and 2 per cent. carbon monoxide was passed through the apparatus, the porcelain tubes being heated to 1100° without any introduction of carbon. One litre of the issuing gas was concentrated over palladium to 58 c.c. The methane found in the residual gas was equivalent to 0.03 per cent. on the original gas collected.

(3) A litre of the same mixture of 98 per cent. hydrogen and 2 per cent. carbon monoxide was, without having been passed through the apparatus, concentrated to 65 c.c. over palladium. The methane found in the residual gas was equivalent to 0.02 per cent. on the original gas collected.

From (2) and (3) it is clear that the presence of even quite a large percentage of carbon monoxide could have no appreciable influence on the production of methane either in the hot tube or during the subsequent concentration over palladium. Any carbon monoxide produced in the actual experiments did not exceed 0.2 to 0.3 per cent.

Actual Experiment.—Total duration=twenty-five hours. Temperature=1100°. Weight of carbon taken=0.086 gram. Total hydrogen used=23 litres.

Sample.	Hour from beginning.	1 litre collected, in minutes.	1 litre concentrated to c.c.	Percentages in the litre of gas collected.		
				CH ₄ .	CO.	N ₂ .
I.....	0—1	65	45	0.93	nil	0.28
II.....	4—5		41	0.51	„	0.75
III.....	10—11		31	0.43	„	0.56
IV.....	12—13		43	0.49	„	0.75
V.....	15—16		60	0.48	„	—
VI.....	19—20		60	0.24	„	—
VII.....	24—25		60	0.20	„	—

Summary.—Estimate of total methane in the issuing gases=102 c.c., of which not more than 2.3 c.c. would be originally contained in the 23 litres of hydrogen used, and not more than 0.4 c.c. could possibly have been derived from impurities in the carbon. Hence 99 c.c. must have been formed by the action of the hydrogen on the carbon, which was still proceeding when the experiment was stopped.

General Summary.—In the above three experiments the formation of no less than 284 c.c. of methane from 0.258 gram of carbon was demonstrated. Some methane was also lost by oxidation or decomposition in contact with the hot walls of the porcelain tube, which became coated internally with a dark brown to black film of either carbon or reduction products.* The exterior of the tube, which had been exposed to the action of the pure hydrogen passing through the annular space between the inner and outer tubes, remained quite white, proving that the reduction of the interior of the tube was due to the action of methane.

D. Experiments showing a 73 per cent. Yield of Methane from less than 0.1 gram of highly purified Carbon at 1150°.

In order to minimise the loss of methane by oxidation in contact with the hot walls of the porcelain tube, the latter was furnished with a loosely-fitting quartz lining, which extended throughout the whole of the heated portion of tube. With the apparatus so

* Since the above paragraph was written, Dr. M. E. Feilmann very kindly undertook a micro-chemical examination of the inner surface of the tube, finding unmistakable signs of lead, but no carbon. It would therefore appear that the methane had reduced some lead compound present in the glaze, with consequent production of metallic lead and carbon monoxide.—W. A. B.

arranged an attempt was made to convert about 0.1 gram of highly purified carbon wholly into methane as follows.

The carbon, enclosed in a piece of platinum foil, was inserted in the middle of the quartz lining of the inner porcelain tube of the apparatus, and heated in a stream of pure dry hydrogen for about thirty-six hours continuously.* The whole of the issuing gas was collected in several successive portions, each of which represented, as a rule, the results of a six hours' run. The volume of each portion having been determined, a litre of it was subsequently concentrated over palladium, the methane being finally determined in the concentrate.

These experiments imposed considerable physical strain upon the operators, and tried the apparatus to the utmost of its capabilities of withstanding the long exposure to the fierce heat of the furnace. The yields of methane obtained, however, approached 75 per cent. of those theoretically possible, and would certainly have been higher had it been possible to prevent all loss by reduction of the inner surface of the porcelain tube. For despite the protection of the loosely-fitting quartz lining, the glaze of the porcelain did not escape attack by the methane. The carbon monoxide in the exit gases, before they had passed through the worms containing ammoniacal cuprous chloride, was found to be about 0.15 per cent., sufficient to account for the whole of the original carbon not actually obtained finally as methane.

Purification of the Carbon.—A new sample of sugar-charcoal was prepared; it was purified in a stream of dry chlorine at 700° for forty-four hours, afterwards at 1100° for thirty-seven hours, and finally in pure dry hydrogen at 1100° for twenty-four hours, until all traces of chlorine were eliminated. It may be of interest to record the results of this drastic treatment, which show how difficult it is to purify carbon completely. After the first forty-four hours in chlorine at 700°, the hydrogen left amounted to 0.5 per cent., and after seventeen more hours at 1100°, it was reduced to 0.1 per cent. At this stage about 0.6 gram was reserved for the final twenty hours' purification in chlorine (which must surely have reduced the hydrogen to practically zero), and an estimate of the methane formed during the twenty-four hours' heating in dry hydrogen, which immediately followed the chlorine treatment, amounted to no less than 160 c.c.

During the final treatment in chlorine some pieces of platinum

* The current of hydrogen was stopped during the short initial period in each experiment during which the tube was being heated, and only resumed fifteen minutes after the attainment of the maximum temperature, when the collection of the first portion of the issuing gas began. In the tabulated results, the various time intervals are reckoned from this point in each case.

wire were mixed with the carbon in order to increase its activity by association with finely-divided platinum; in this way, about 11 per cent. of platinum was taken up.

The details of two experiments are tabulated below.

Experiment I.*

Total duration after attainment of maximum temperature = thirty-seven hours continuously. Temperature = 1150° . Total carbon used = 0.0995 gram. Hydrogen used = about 34 litres, which was dried by means of calcium chloride and solid potassium hydroxide.

Period	1	2	3	4	5	6	7
Hour	0 to 6	6 to 12	12 to 18	18 to 24	24 to 30	30 to 36	36 to 37
Total volume of gas collected, in litres	5.6	5.5	5.5	5.5	5.5	5.6	1.0
Percentages in gas collected							
CO ...	0.025	0.02	0.03	0.01	0.03	0.02	nil
CH ₄ ...	0.65	0.57	0.59	0.56	0.26	0.06	0.91
N ₂ ...	[3.6]	0.07	0.25	0.10	0.20	0.05	0.05

Total yield of methane = 148 c.c. at 22° and 752 mm. = 135.5 c.c. at 0° and 760 mm.

Theoretical yield from 0.0995 gram of carbon = 185 c.c. at 0° and 760 mm.

\therefore Actual yield = 73.3 per cent. of that theoretically possible.

Experiment II.

Total duration after attainment of maximum temperature = thirty-five hours continuously. Temperature = 1120 – 1180° .

Carbon used = 0.092 gram. Hydrogen used = 32.6 litres.

Throughout the whole of this experiment, the hydrogen, after being dried by means of calcium chloride and solid potassium hydroxide as previously described, was passed through the spiral *F* (Fig. 2) externally cooled with liquid air, and it therefore entered the porcelain tube containing the carbon in a perfectly dry condition. The pure hydrogen passed through the annular space

* Before this experiment, a "blank" was performed, in which pure dry hydrogen was passed through the apparatus with the porcelain tube heated to 1150° , but containing no carbon. 970 c.c. of the issuing gas were collected and reduced to 18 c.c. over palladium foil. The methane found in the residue corresponded with 0.01 per cent. on the original gas collected, a negligible quantity.

CARBON AND HYDROGEN. SYNTHESIS OF METHANE. 1991

between the two tubes was also similarly dried by passage through spiral immersed in liquid air.

Period	1	2	3	4	5	6	7
Hour	0 to 6	6 to 12	12 to 18	18 to 24	24 to 30	30 to 34	34 to 35
Total volume of gas collected, in litres	5.62	5.60	5.62	5.70	5.60	3.50	1.00
Room temperature	21°	22°	21.5°	22°	22°	22°	—
Barometer	757	758	758	759	758.5	758	—
Percentages in gas collected	$\begin{Bmatrix} \text{CO} \dots & 0.02 \\ \text{CH}_4 \dots & 0.65 \\ \text{N}_2 \dots & 0.07 \end{Bmatrix}$	$\begin{Bmatrix} 0.01 \\ 0.66 \\ 0.09 \end{Bmatrix}$	$\begin{Bmatrix} 0.01 \\ 0.58 \\ 0.08 \end{Bmatrix}$	$\begin{Bmatrix} 0.00 \\ 0.41 \\ 0.06 \end{Bmatrix}$	$\begin{Bmatrix} 0.00 \\ 0.11 \\ 0.07 \end{Bmatrix}$	$\begin{Bmatrix} 0.01 \\ 0.02 \\ 0.25 \end{Bmatrix}$	$\begin{Bmatrix} 0.02 \\ 0.018 \\ 0.15 \end{Bmatrix}$

Total yield of methane = 136 c.c. at 22° and 758 mm. = 125.5 c.c. at 1° and 760 mm.

Theoretical yield of methane from 0.092 gram of carbon = 172 c.c. at 1° and 760 mm.

∴ Actual yield = 73.1 per cent. of that theoretically possible.

Experiment III.

Total duration after attainment of maximum temperature = eighteen hours. Temperature = about 1100°. Carbon used = 0.09 gram. Hydrogen used = 16.5 litres (dried by solid potassium hydroxide).

The apparatus unfortunately broke down after eighteen hours and before all the carbon was used up; during the eighteen hours' run, 108 c.c. of methane were obtained as follows.

Period	1	2	3
Hours	0 to 6	6 to 12	12 to 18
Total volume of gas collected (litres)	5.5	5.5	5.5
Percentages in the gas collected	CO	0.02	0.21
	CH ₄	0.67	0.44
	N ₂	0.19	1.85

Experiment IV.

In this experiment a very much smaller quantity of carbon, 0.0247 gram only, was employed; the hydrogen was dried over calcium chloride and solid potassium hydroxide. The methane formed was 28.1 c.c. at 0° and 760 mm., or a yield of 61 per cent. of the 46.1 c.c. theoretically possible, a greater proportion being lost by oxidation than in the previous experiments.

General Summary.—In the above four experiments we have proved the formation of 422 c.c. of methane, measured at about 20–22° and at atmospheric pressure, from a total of 0.3062 gram of carbon, which corresponds with 68.5 per cent. of the yield theoretically possible (616 c.c.). In two experiments the yield was 73 per cent. of that theoretically possible, and in one of these experiments the hydrogen, after being passed over calcium chloride and solid potassium hydroxide, was perfectly dried by passage through a long spiral immersed in liquid air. In addition to the above yield of methane, another 160 c.c. were formed during the twenty-four hours' heating in dry hydrogen which intervened between the final purification of the 0.6 gram of carbon in chlorine and the commencement of experiment I, so that altogether the formation of about 580 c.c. of methane was demonstrated.

In our opinion these results prove beyond question that methane is formed at 1100° to 1200° by the direct action of hydrogen on carbon, as originally stated by Bone and Jerdan in 1897.

E. Note on the Increase in the Activity of Sugar-charcoal towards Hydrogen, due to Admixture with Platinum.

Reference has already been made to the increase observed in the activity of the purified sugar-charcoal used in the foregoing experiments (sections C and D), due to its previous admixture with from 10 to 15 per cent. of finely-divided platinum. From the point of view of the main object of our experiments, namely, to convert a small quantity of carbon wholly or almost wholly into methane, this was a fortunate circumstance, inasmuch as it doubtless shortened the time required, which otherwise would probably have exceeded the limits of endurance both of the apparatus and the operators. The degree of stimulation due to the platinum may be judged from the two following strictly comparable experiments, in which exactly equal quantities of the same highly-purified sugar-charcoal (containing less than 0.1 per cent. of hydrogen), in the one case with and in the other case without addition of platinum, were heated in pure dry hydrogen to 1150° and the issuing gas collected during

the first hour after the attainment of the maximum temperature, thus:

	Carbon + 11 per cent. of platinum.	Carbon without platinum.
Weight of carbon taken.....	0.105 gram	0.105 gram
Volume of gas collected.....	1000 c.c.	970 c.c.
Percentage of CH ₄ formed.....	0.75	0.35

In the case of the carbon unmixed with platinum, an increase in the methane formed was effected by merely increasing the weight of carbon used; thus with 0.6 gram of carbon as much as 0.65 per cent. of methane was obtained. With a slow action of this kind, the amount of methane obtained might be expected to increase with the surface area of the carbon exposed to the action of the hydrogen, although not necessarily proportionately, until the equilibrium point of the system $C + 2H_2 \rightleftharpoons CH_4$ was attained. The nearer the approach to the equilibrium point the less would a given increase in the surface of the carbon affect the percentage of methane obtained.

In conclusion, we desire to thank the Government Grant Committee of the Royal Society for grants, out of which the expenses of these experiments have been partly met.

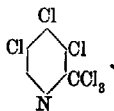
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XCVII.—*The Chlorination of Methyl Derivatives of Pyridine. Part II. 2-Methylpyridine.*

By WILLIAM JAMES SELL.

As a former communication (Part I, Trans., 1905, 87, 799) it was shown that, when α -picoline was first saturated with hydrogen chloride and then subjected to chlorination at 105–110° as long as any action appears to take place, a mixture of chlorinated derivatives was obtained, from which a solid compound, shown to be a hexachloro- α -picoline, was separated. This, on examination, proved to have the constitution represented by the formula:



It was further shown (*loc. cit.*) that when this compound is carefully heated with 80 per cent. sulphuric acid it is converted into 3:4:5-trichloropicolinic acid, which is deposited when the cooled liquid is diluted with water, any 3:4:5-trichloropyridine formed by local over-heating being retained in solution.

Examination of the Liquid Product of the Chlorination of α -Picoline.

The liquid, which was drained from the crystals of hexachloropicoline by the aid of the pump and pressure, was submitted to fractional distillation under reduced pressure. The liquid commenced to distil at 148°/12 mm., the thermometer quickly rising to 153°; the receiver was changed every 5° until the thermometer registered 183°, when the distillation was practically complete. By far the largest part of the distillate passed over between 153° and 168° and remained liquid, whereas the fractions boiling at 168—183° showed signs of crystallisation on cooling, and, after some months, became almost solid. The solid from each of the last three fractions was collected, and, on crystallisation from alcohol, was found to separate in the characteristic prisms of the hexachloropicoline, m. p. 102—103°, already described, and this conclusion was confirmed by heating it with 80 per cent. sulphuric acid, when trichloropicoline, m. p. 164—165°, was isolated. That hexachloropicoline is freely soluble in the liquid products was well known, and it is not therefore surprising to find it distilling over in the last part of the distillation. Its boiling point under 12 mm. pressure is 175—180°.

In the hope of effecting some further purification by freezing out, draining, and crystallisation, the various liquid fractions were immersed in ice and then in a mixture of ice and salt, with no other result than to render them thick and syrupy. When portions were next gradually exposed in the upper part of a vessel containing liquid air, they remained clear for a time, and of the consistency of Canada balsam, ultimately becoming opaque and solid. On removal from the bath, the phenomena were repeated in the inverse order, and, after many fruitless attempts, this method of purification was abandoned.

Examination of the Various Fractions by heating with 80 per cent. Sulphuric Acid.

Portions of each fraction, b. p. 153—168°, weighing 20 grams, were mixed with 50 c.c. of 80 per cent. sulphuric acid, and heated to 130—132° in a long-necked flask provided with an air condenser ground into its neck, the heating being continued until fumes of hydrogen chloride ceased to escape. The mixture, on cooling, was next diluted with about 200 c.c. of water, and distilled in a current of

steam, when generally a few drops of apparently unaltered compound passed over, and the liquid in the flask deposited a crop of crystals on cooling.

The crystals were collected and purified by recrystallisation from water, when they were deposited in colourless needles. Those crystals which separated from the treatment of the fractions boiling at 153—158° and 158—163° melted with decomposition at 153—154°, whereas the acid obtained from the still liquid portions of the higher fractions melted at 148—149° under similar treatment as to purification. Subsequent further purification by recrystallisation, however, raised the temperature of the acid from the later fractions to 153—154°, and, as was expected and confirmed by qualitative tests, the depression was due to the crystals being more or less contaminated with trichloropicolinic acid derived from hexachloropicoline contained in the fractions of higher boiling point.

3:5-Dichloropicolinic acid is fairly soluble in boiling, and sparingly so in cold, water, freely so in boiling alcohol and in most organic solvents. The crystals are anhydrous, and gave the following numbers on analysis:

0.1855 gave 0.255 CO₂ and 0.028 H₂O. C = 37.48; H = 1.67.

0.3225 „ 0.4770 AgCl. Cl = 36.60.

0.0985 „ 0.1460 AgCl. Cl = 36.88.

C₆H₃O₂NCl₂ requires C = 37.50; H = 1.56; Cl = 36.97 per cent.

An aqueous solution of the acid gives the following reactions:

(1) With calcium or barium chlorides and a drop of ammonia, no precipitate.

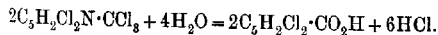
(2) With silver nitrate, a sparingly soluble, flocculent precipitate consisting of woolly needles.

(3) With mercuric chloride, a precipitate which is slowly deposited in stout, colourless needles.

(4) With ferrous sulphate, a faint brown coloration and, after long standing, a slight dark red, crystalline deposit.

(5) With copper acetate, a light blue, sparingly soluble copper salt.

The foregoing results show that a large part of the chlorination product of α -picoline, which is liquid at the ordinary temperature, consists of pentachloropicoline, C₅H₂Cl₂N·CCl₃, and that this on heating with 80 per cent. sulphuric acid, is resolved into dichloropicolinic acid and hydrogen chloride, thus:



It may be mentioned that some of the compounds described in this series of papers are isomeric with the corresponding substances obtained by Ost by the action of phosphorus pentachloride on cemenamic acid (*J. pr. Chem.*, 1883, [ii], 27, 257).

Methyl-3 : 5-dichloropicolinate.

About 40 grams of the acid were converted into its methyl ester in the usual way, the substance being recrystallised from methyl alcohol. It forms stout needles, melting at 78—79° (uncorr.), and may be crystallised from water or distilled in a current of steam without change, as shown by the melting point of the material after these operations.

3 : 5-Dichloropicolinamide.

The greater part of the methyl ester was treated with an excess of strong aqueous ammonia (0.880) and left overnight in a closed flask. The amide was dissolved in boiling water, from which, on cooling, it crystallised in colourless needles melting sharply at 175—176° (uncorr.):

0.1795 gave 0.2455 CO₂ and 0.0345 H₂O. C = 37.29; H = 2.13.

0.2593 „ 32.15 c.c. N₂ at 17.9° and 767.5 mm. N = 14.57.

C₆H₄O₂N₂Cl₂ requires C = 37.69; H = 2.09; N = 14.66 per cent.

Conversion of 3 : 5-Dichloropicolinic Acid into 3 : 5-Dichloropyridine.

Since the effervescence occasioned by heating the acid to its melting point seemed to indicate that it loses carbon dioxide at this temperature, an experiment was arranged which consisted in distilling about 5 grams of the acid with 20 c.c. of glycerol. The distillate, which on cooling solidified to a crystalline mass, was rinsed with cold alcohol and after pressure between folds of blotting paper crystallised from hot alcohol, from which it separated in flat, flexible needles and plates with the characteristic odour, volatility, reactions, and melting point (66—67°) of the dichloropyridine described by Königs and Geigy (*Ber.*, 1884, 17, 589, 1833), Sell and Dootson (*Trans.*, 1899, 79, 979), and shown by the author to contain the chlorine atoms in the 3 : 5-position (this vol., 1997). This was confirmed by converting a specimen of the dichloropyridine into diethoxypyridine and transforming the latter by bromine water into the characteristic dibromodiethoxypyridine, m. p. 193—140°:

Found, Br = 49.29

C₆H₁₁O₂NBr₂ requires Br = 49.23 per cent.

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XVIII.—*The Chlorine Derivatives of Pyridine.*
 Part IX. *Preparation and Orientation of 3:5-Dichloropyridine.*

By WILLIAM JAMES SELL.

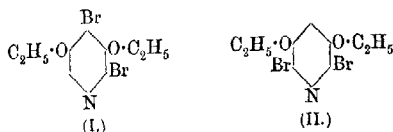
THE substance which forms the subject of this communication has long been known, and was first described by Königs and Geigy, who obtained it together with a trichloropyridine by the interaction of phosphorus pentachloride and barium pyridinedisulphonate (*Ber.*, 1884, 17, 589, 1833). The dichloropyridine has since been obtained by Dootson and the author (*Trans.*, 1899, 79, 979), and, more recently, the author has found the yield of this substance much improved by modifying the apparatus previously used (*loc. cit.*) so as to encourage the sublimation of the dichloropyridine hydrochloride as the chlorination proceeds. The same compound was also found to result from the distillation with glycerol of the dichloropicolinic acid prepared by the action of 80 per cent. sulphuric acid on one of the products of the chlorination of α -picoline (this vol., 1993).

The well-marked basic characters of the substance, together with its great stability, as indicated especially by its unalterability by ammonia at any temperature below 300°, suggested that the chlorine atoms in the compound occupied the 3:5-positions. This conclusion was strengthened by the analogy in general properties and stability under similar conditions to the dibromopyridine, m. p. 110–111°, in which the bromine atoms are known to occupy the 3:5 positions. The latter substance was discovered by Hofmann (*Ber.*, 1879, 12, 988), and the orientation conclusively proved by, among others, G. Pfeiffer (*Ber.*, 1887, 20, 1343).

With the object of testing the above conjecture, it was determined to prepare a quantity of Hofmann's 3:5-dibromopyridine, m. p. 110–111°, according to the directions of that investigator (*loc. cit.*). It was then converted into the diethoxypyridine by heating in sealed tubes with excess of sodium ethoxide.

Similarly, a quantity of diethoxide was made from the dichloropyridine in question. The examination of the two specimens of diethoxypyridine showed that they were similar to each other, and corresponded with the description given by Weidel and Blau (*Monatsh.*, 1885, 6, 669), who prepared their specimen from 3:5-dibromopyridine. It was then determined to institute experiments in the hope of obtaining some sparingly soluble, definite, crystalline compound of marked individuality, by which a comparison could be made of the

identity of the diethoxide from the two sources. The result was the discovery that the diethoxide from both sources reacts with bromine and chlorine to form identical compounds. When bromine water is added to an aqueous solution of the diethoxide in dilute hydrochloric acid, a bulky, white precipitate is produced, consisting of delicate needles almost insoluble in water, but fairly soluble in dilute alcohol, from which the substance crystallises in somewhat flattened needles with a satiny sheen, melting at 141—142° (uncorr.). As will be seen below, the diethoxypyridine absorbs two atoms of bromine, and in view of the fact that by heating the compound to 150° with concentrated hydrochloric acid it is converted into dibromodihydroxypyridine, the new compound is represented by one of the subjoined formulæ, of which (I) is regarded as the more probable, but further experiments are necessary to justify it.



An analogous compound containing two atoms of chlorine is obtained from the diethoxide from both sources by the use of chlorine water in place of bromine water. The compound forms beautiful needles from dilute alcohol, melting at 123—124°. This substance, when heated with concentrated hydrochloric acid to 150°, is converted into dichlorodihydroxypyridine, crystallising from water in colourless needles containing 3 molecules of water of crystallisation, which are lost in a vacuum over sulphuric acid, the substance becoming opaque. The compound is represented by one of the following formulæ, but the question as to which is the correct one must be the subject of further study.



EXPERIMENTAL.

The dichloropyridine (2 grams) was heated in sealed tubes for five hours with a solution of sodium (1.5 grams) in absolute alcohol (20 c.c.) to a temperature of 190—200°. After cooling, the contents of the tubes were rinsed out with water, and distilled in a current of steam. The first part of the distillate consisted chiefly of alcohol with some dissolved diethoxypyridine, and this was followed by an

aqueous part which separated into two layers, the lower of which was mainly diethoxypyridine, which was separated in the usual way, dissolved in dilute hydrochloric acid, and used for the experiments detailed below.

A small part was precipitated with chloroplatinic acid, and a platinumchloride, consisting of orange-yellow needles, was obtained agreeing with the description given by Weidel and Blau (*loc. cit.*). The substance is unchanged on heating to 100°, and is anhydrous. Two portions which had been heated to 100° were afterwards ignited, and gave the following results:

0.418 gave 0.110 Pt. Pt = 26.31.

0.3278 „ 0.0855 Pt. Pt = 26.08.

$[C_9H_5(OEt)_2N]_2, H_2PtCl_6$ requires Pt = 26.18 per cent.

Conversion of Diethoxypyridine into Dibromodiethoxypyridine.

A further portion of diethoxypyridine hydrochloride was mixed with bromine water, when a voluminous, white precipitate was produced consisting of colourless needles. These were practically insoluble in cold water, and only slightly soluble on boiling, but readily so in hot alcohol, from which they crystallised in somewhat flattened needles, having a pearly lustre and melting at 141–142° (uncorr.):

(1) 0.2077 gave 0.2562 CO_2 and 0.0705 H_2O . C = 33.60; H = 3.77.

(2) 0.2126 „ 0.2618 CO_2 „ 0.0665 H_2O . C = 33.58; H = 3.48.

(3) 0.127 „ 0.142 AgBr. Br = 48.75.

(4) 0.1228 „ 0.142 AgBr. Br = 49.23.

$C_9H_{11}O_2NBr_2$ requires C = 33.23; H = 3.39; Br = 49.23 per cent.

Conversion of Diethoxypyridine into Dichlorodiethoxypyridine.

Another part of the diethoxypyridine hydrochloride was precipitated with chlorine water, the bulky, crystalline precipitate collected, and recrystallised from dilute alcohol. The substance forms beautiful colourless needles when the solution is allowed to cool rapidly, but when cooled slowly, large, prismatic crystals are formed, resembling those of potassium nitrate, and melting at 124° (uncorr.). The substance dissolves only sparingly in water, but is freely soluble in alcohol, ether, or acetone:

0.1795 gave 0.2176 AgCl. Cl = 29.97.

$C_9H_{11}O_2NCl_2$ requires Cl = 30.08 per cent.

Formation of Dibromodiethoxypyridine from 3:5-Dibromopyridine.

Fifty grams of pure dibromopyridine, m. p. 111°, prepared by Hofmann's directions (*loc. cit.*), were treated in an exactly similar

manner to the dichloropyridine, and part of the diethoxypyridine was converted by bromine water into its bromine derivative. The crystals formed were indistinguishable in appearance, solubility, and melting point (141—142°) from the specimen obtained by similar treatment of the dichloropyridine in question :

0.0915 gave 0.10574 AgBr. Br = 49.17.

0.0963 „ 0.11144 AgBr. Br = 49.24.

$C_6H_{11}O_2NBr_2$ requires Br = 49.23 per cent.

The remaining diethoxypyridine, prepared from 3 : 5-dibromopyridine, was converted by means of chlorine water into the dichlorodiethoxypyridine. The specimen thus produced was indistinguishable from that prepared from the dichloropyridine in question.

Conversion of Dichlorodiethoxypyridine into Dichlorodihydroxypyridine.

Four grams of dichlorodiethoxypyridine were heated to 150—155° with concentrated hydrochloric acid in sealed tubes, each tube being charged with 1 gram of the substance and 20 c.c. of acid. The heating was continued for six hours, and if, on cooling, the tube is free from crystalline matter, the decomposition is complete. On opening the tubes, ethyl chloride escaped, and on evaporating the contents of the tubes to dryness on the water-bath, a slightly brown residue was obtained. This was dissolved in the least possible amount of boiling water, a small quantity of animal charcoal added, and, after digestion for a time, the solution was filtered and allowed to crystallise. The substance separates from water in fine, colourless needles containing 3 molecules of water of crystallisation, which it loses in a vacuum over sulphuric acid, or in the air-bath at 60—70°.

The undried compound when heated to 83—84° partly melts, leaving a lower hydrate, or more probably the unmelted anhydrous substance. On continuing the heating, the whole solidified, and afterwards melted with darkening at 196° (uncorr.). The substance when dried at 60—70° melts sharply at 194—195° (uncorr.) to a colourless liquid :

0.2105 lost, in a vacuum over sulphuric acid, 0.0485. $H_2O = 23.04$.

0.0835, dry substance, gave 0.13312 AgCl. Cl = 39.41.

$C_5H_3O_2NCl_2 \cdot 3H_2O$ requires $H_2O = 23.07$ per cent.

$C_5H_3O_2NCl_2$ „ Cl = 39.44 „

UNIVERSITY LABORATORY,
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CXCIX.—*The Chlorine Derivatives of Pyridine. Part X.*
Orientation of 2 : 3 : 5-Trichloropyridine.

By WILLIAM JAMES SELL.

THIS compound was first described by Königs and Geigy, who obtained it, together with a dichloropyridine, by the interaction of phosphorus pentachloride and barium pyridine disulphonate (*Ber.*, 1884, 17, 589, 1833).

It was also obtained in relatively large amount by Dootson and the author by the interaction of phosphorus pentachloride and pyridine (*Trans.*, 1898, 81, 432), as well as by the chlorination of pyridine hydrochloride. In the latter case, it was separated by fractionation, and cooling of the products boiling at 205—221°/762 mm.

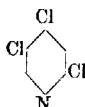
Orientation.

Königs and Geigy found (*loc. cit.*) that, by the action of sodium ethoxide on this compound, one of its chlorine atoms was readily replaced by ethoxyl, and, from the similar ease with which the chlorine is replaced in 2-chloroquinoline, they were inclined to consider that one of the chlorine atoms in this trichloropyridine was also in the 2-position. The conclusion happens to be correct, but the reasoning is weak and calculated to lead to erroneous conclusions, as, for example, in the case of 2:3:4:5-tetrachloropyridine, where the chlorine atom occupying the 4-position is the one most easily removed by sodium ethoxide (*Trans.*, 1903, 83, 396).

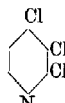
Excluding the trichloropyridines of known constitution, and regarding the feeble, but distinct, basic properties of the one in question, there was every probability that its constitution would be represented by one of the following formulæ :



(I.)



(II.)

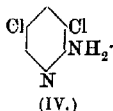


(III.)

of which (I) was regarded as sufficiently probable to warrant the attempt to determine its orientation on the following lines.

A compound having this formula ought to be attacked by ammonia and give 3:5-dichloro-2-aminopyridine, having the constitution re-

presented by (IV). This was carried out, and a substance melting at 84—85° was isolated :



Further, 3 : 5-dichloropicolinamide (this vol., 1996) was converted by the Hofmann reaction into 3 : 5-dichloro-2-aminopyridine, represented by (IV) above. The specimen thus obtained melted at 84—85°, and the melting point was unaffected by admixture with the specimen prepared by the action of ammonia on the trichloropyridine melting at 49—50°.

Moreover, Königs and Geigy have described (*loc. cit.*) a dichloro-ethoxypyridine obtained from this trichloropyridine by the action of sodium ethoxide. This compound they afterwards converted by heating with hydrochloric acid to 150° into the corresponding dichloro-hydroxypyridine, m. p. 178°.

Finally, the compound depicted above (IV), obtained through dichloropicolinamide by the Hofmann reaction, is converted by a modification of the Bouveault reaction into the same dichlorohydroxypyridine, m. p. 178—179°, as was obtained from the trichloropyridine, m. p. 49—50°.

EXPERIMENTAL.

Formation of 3 : 5-Dichloro-2-aminopyridine from Trichloropyridine, m. p. 49—50°.

Three tubes, each containing 2 grams of trichloropyridine, m. p. 49—50°, dissolved in a mixture of equal volumes of alcohol and concentrated aqueous ammonia, were sealed and heated to 180° for three hours. The contents of the tubes were evaporated to remove most of the alcohol, diluted with water, and, after cooling, the precipitated compound was collected and washed. The substance thus obtained was treated with dilute hydrochloric acid, when a small quantity of the unaltered compound remained. The filtrate was precipitated by ammonia, distilled in a current of steam, and the amino-compound was recrystallised from hot dilute alcohol, from which it separates in needles or prisms melting at 84—85°. The compound is freely soluble in alcohol or acetone, moderately so in hot water, but very sparingly so in the cold liquid. It dissolves readily in dilute acids. The solution in dilute hydrochloric acid gives a *platinichloride*, which separates in orange-yellow prisms containing 2 molecules of water of crystallisation :

0.193 gave 0.1095 CO_2 and 0.0325 H_2O . $\text{C} = 15.47$; $\text{H} = 1.87$.

0.3825 lost, at 100° , 0.0185 and, on ignition, gave 0.0965 Pt.

$\text{H}_2\text{O} = 4.62$; $\text{Pt} = 25.22$.

$\text{H}_2\text{N}_4\text{Cl}_4, \text{H}_2\text{PtCl}_6, 2\text{H}_2\text{O}$ requires $\text{C} = 15.54$; $\text{H} = 1.806$; $\text{H}_2\text{O} = 4.66$;
 $\text{Pt} = 25.23$ per cent.

Preparation of 3:5-Dichloro-2-aminopyridine from 3:5-Dichloropicolinic Acid.

Four grams of 3:5-dichloropicolinamide were submitted to the Hofmann reaction, the final distillation from the strongly alkaline solution being omitted as being unnecessary and likely to cause loss of chlorine from the compound.

The 3:5-dichloro-2-aminopyridine which separated was collected, washed, and further purified by solution in hydrochloric acid and precipitation by ammonia. It forms colourless crystals, melting at $84-85^\circ$, giving a similar platinichloride, and having properties in complete agreement with those of the sample of the same substance prepared from the trichloropyridine, m. p. $49-50^\circ$, in solution.

Preparation of 3:5-Dichloro-2-hydroxypyridine from 3:5-Dichloro-2-aminopyridine.

Two grams of 3:5-dichloro-2-aminopyridine were dissolved in 10 per cent. sulphuric acid, mixed with a small excess of nitrophenolic acid in sulphuric acid, and warmed on the water-bath for about half an hour. The mixture was then poured into cold water, and the whole boiled for two or three minutes, cooled, and nearly neutralised with sodium hydroxide, when a precipitate was obtained from the still acid solution. The substance was collected, washed, and recrystallised from water, from which it separates in needles, m. p. $178-179^\circ$.

In conclusion, the author desires to acknowledge the valuable assistance rendered by Mr. G. Hall in the preparation of material for this and other researches on the chloropyridines. His grateful thanks are also due to the Government Grant Committee of the Royal Society for defraying the chief expenses of these researches.

UNIVERSITY LABORATORY,
CAMBRIDGE.

CC.—Organic Derivatives of Silicon. Part VII.
The Synthesis of *dl*-Sulphobenzylethylisobutylsilicyl
Oxide.

By BERNARD DUNSTAN WILKINSON LUFF, A.I.C. (1851 Exhibition Scholar), and FREDERIC STANLEY KIPPING.

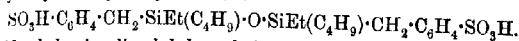
UP to the present time the only silicon compound which has been resolved into its optically active components is the sulphonie derivative of *dl*-benzylethylpropylsilicyl oxide, $[\text{SO}_3\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{EtPrSi}]_2\text{O}$, an acid which contains two asymmetric silicon groups. Attempts to resolve other *dl*-silicon derivatives which contain only one asymmetric group have so far been unsuccessful; the fruitless experiments made in the case of *dl*-benzylmethylethylpropylsilicanesulphonic acid, $\text{MeEtPrSi}\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}$, have already been recorded (Kipping, Trans., 1907, 91, 717), and several similar investigations which have been in progress in these laboratories during the past year have also failed to attain the end in view. Whether work on these lines is ultimately brought to a successful issue or not, it seemed very desirable to extend and corroborate our knowledge of optically active silicon derivatives by preparing and studying at least a second example of a compound containing two asymmetric silicon groups; all the more so in view of the fact that the specific rotations of the *d*- and *l*-sulphobenzylethylpropylsilicyl oxides are very small, and in other respects also the behaviour of the active compounds is not very clearly indicative of their enantiomorphous relationship.

With this object in view, we began experiments on the synthesis of *dl*-benzylethylisobutylsilicol, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\cdot\text{OH}$, in the expectation that the behaviour of this compound would be, on the whole, very similar to that of its lower homologue, and that it would yield a sulphonie derivative of *dl*-benzylethylisobutylsilicyl oxide which might be resolved into its *d*- and *l*-components; in this paper we describe the results of these experiments and the properties of the various compounds which have been obtained.

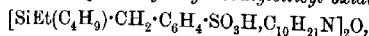
Starting from ethylsilicon trichloride, we first prepared benzyl-ethylsilicon dichloride, but under conditions different from those employed and described previously (Trans., 1907, 91, 720). This compound was then treated with magnesium isobutyl iodide in ethereal solution, but the results were highly unsatisfactory. By employing magnesium isobutyl bromide instead of the iodide, although the product was a complex mixture, we were able to isolate from it *dl*-benzylethylisobutylsilicyl chloride, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\text{Cl}$, as a fuming oil, boiling at $198-202^\circ/100$ mm. By decomposing *dl*-benzylethylisobutylsilicyl chloride with aqueous ammonia, we obtained *dl*-benzylethylisobutylsilicol, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\cdot\text{OH}$, as a colourless oil, boiling at $198-202^\circ/100$ mm.

butylsiliclyl chloride with water or with sodium carbonate solution, we obtained *dl*-benzylethylisobutylsiliclyl, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\cdot\text{OH}$, and *dl*-benzylethylisobutylsiliclyl oxide, $[\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)]_2\text{O}$, two compounds which were easily separated by fractional distillation, as the former boils at $162\text{--}164^\circ/25\text{ mm.}$, and the latter at $250\text{--}252^\circ/20\text{ mm.}$

Experiments were then made in order to find a satisfactory method for the preparation of a sulphonic acid from one of the three *dl*-compounds just mentioned, but the task proved to be unexpectedly difficult; ultimately, from the sulphonation products of the siliclyl, the oxide, and the chloride, we obtained, in the form of its *l*-menthylamine salt, a *dl*-sulphonic acid which proved to be a derivative of sulphonylbenzylethylisobutylsiliclyl oxide,

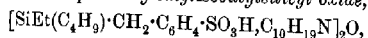


l-Menthylamine *dl*-sulphonylbenzylethylisobutylsiliclyl oxide,



crystallises with 4 molecules of water, melts at $240\text{--}245^\circ$ and its specific rotation in methyl-alcoholic solution is $[\alpha]_D - 14.7^\circ$. It is very similar to the corresponding derivative of benzylethylpropylsiliclyl oxide, and, like the latter, it is not resolved into its components by fractional crystallisation from aqueous alcohol or moist ethyl acetate. This compound was used merely as a means of isolating the pure *dl*-acid from the mixture of products obtained in the process of sulphonation. The sodium salt prepared from it is crystalline and readily soluble in water; the barium salt is practically insoluble in water and in alcohol, but dissolves readily in a mixture of these solvents.

dl-Bornylamine *dl*-sulphonylbenzylethylisobutylsiliclyl oxide,



crystallises from aqueous alcohol in needles, and melts at $207\text{--}209^\circ$.

Cinchonidine dl-sulphonylbenzylethylisobutylsiliclyl oxide crystallises in needles, melts at $175\text{--}177^\circ$, and its specific rotation in methyl-alcoholic solution is $[\alpha]_D - 69.3^\circ$.

Cinchonidine hydrogen sulphonylbenzylethylisobutylsiliclyl oxide separates from anhydrous acetone containing a trace of methyl alcohol as a microcrystalline powder which melts and decomposes at $225\text{--}229^\circ$.

The results of the experiments on the resolution of the *dl*-acid will be described in the next communication.

EXPERIMENTAL.

Preparation of Benzylethylsilicon Dichloride, $\text{Si}(\text{C}_7\text{H}_7)\text{EtCl}_2$.

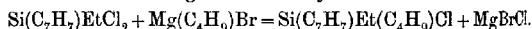
The preparation of this compound has been described in a previous communication (Kipping, *Trans.*, 1907, **91**, 720); the method there involved two operations, namely, the preparation of the Grignard compound and the subsequent addition of this to the silicon deriv-

ative, a process which had to be carried out very slowly. An attempt was therefore made to combine the two reactions in one operation, the magnesium benzyl chloride being formed in the presence of excess of the ethylsilicon trichloride. This method was found to give satisfactory results, and was carried out as follows.

Ethylsilicon trichloride (1 mol.), diluted with about six times its volume of dry ether, and magnesium filings (1 mol.) are placed in a flask, which is fitted with a cork carrying a tap funnel, and also with a stirrer. A small quantity of benzyl chloride is run in, and the contents of the flask are agitated for a moment; if the reaction does not start spontaneously, a small quantity of an ethereal solution of magnesium benzyl chloride is added. When the reaction has once set in, as shown by the liquid becoming warm, the flask is immersed in ice and the contents are kept cool, and vigorously stirred, while the rest of the benzyl chloride (1 mol.) is added. The separation of magnesium chloride sets in almost from the outset, and the reaction seems to be completed at the ordinary temperature. The product is worked up as described previously (see above), and the yield of pure benzylethylsilicon dichloride is about 60—70 per cent. of the theoretical. By this modified method, only a very small quantity of dibenzyl is formed compared with the amount produced when the original process is employed; this is a great advantage, as the separation of this impurity from the desired silicon compound by fractional distillation is a rather troublesome operation.

Benzylethylisobutylsilicyle Chloride, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\text{Cl}$.

This compound was prepared by the interaction of benzylethylsilicon dichloride and magnesium isobutyl bromide:



The ethereal solution of magnesium isobutyl bromide (1 mol.) is added to an ethereal solution of benzylethylsilicon dichloride (1 mol.), contained in a flask immersed in cold water and provided with a stirrer. No appreciable rise in temperature takes place, so that the liquids may be mixed fairly quickly, and no magnesium salt is deposited for some time. In order to hasten the completion of the reaction, the mixture is heated under reflux for six hours on a water-bath. The ether is then evaporated, and the residue heated on the water-bath for about eight hours longer. After these operations, a bulky deposit of magnesium salt is obtained, and the oily product contained in it is extracted by means of ether in an apparatus from which moist air is excluded (Trans., 1907, 91, 216). The ether is then evaporated, whereupon a further separation of magnesium salt usually occurs, which may involve a repetition of the above process.

centually there results a brownish-yellow oil, which is distilled from an ordinary Wurtz flask under a pressure of 100 mm. Practically the whole of this liquid passes over below 230°, but during the distillation a further quantity of magnesium chlorobromide is deposited, and the residue consists partly of this salt and partly of silicon compounds of high boiling point; the quantity of the latter is relatively small. The distillate is then submitted to fractional distillation (at 100 mm.), using a flask with a long neck and employing a rod and disk column. Fractions are collected below 190°, from 190° to 210°, and from 210° to 225°; the largest is that collected between 190° and 210°. In order to obtain the pure chloride, these fractions are systematically redistilled, and the liquid boiling at 196—204° is first collected. This preparation is still impure, and generally contains about 15·3 per cent. chlorine; when it is fractionated again three or four times, it yields a colourless liquid, boiling at 198—202° (100 mm.), which gives satisfactory results on analysis*:

2338 required 18·9 c.c. AgNO_3 solution (1 c.c. = 0·00177 Cl). Cl = 14·8.
 $\text{C}_{13}\text{H}_{21}\text{ClSi}$ requires Cl = 14·7 per cent.

The isolation of the pure chloride is exceedingly troublesome, owing to the presence of unchanged benzylethylsilicon dichloride, and probably so to that of benzylethylisobutylsilicane. For the preparation of the silicol, a fraction boiling from 196° to 202°/100 mm. may be employed; the yield of this fairly pure chloride amounts to about 1 per cent. of the theoretical.

Benzylethylisobutylsilicyl chloride is a colourless, fuming, mobile liquid, readily decomposed by water.

Decomposition of Benzylethylisobutylsilicyl Chloride by Water.

As it seemed very probable that this chloride, like the corresponding benzylethylpropylsilicyl derivative, would be decomposed by water, giving the oxide as well as the silicol, and, further, that the silicol could be the more suitable for the preparation of a sulphonic derivative, we tried to obtain as large a proportion as possible of the silicol by treating the chloride with an ice-cold solution of sodium carbonate (trans., 1908, 93, 461).

The crude product, an almost colourless oil, is submitted to fractional distillation under a pressure of 25 mm., using an ordinary distilling flask. A large fraction passes over at 150—180°, this being fairly pure benzylethylisobutylsilicol; the temperature then rises rapidly to 240°, and another large fraction, which contains the corresponding chloride, passes over at 240—280°; the flask then usually contains a

* The analysis was carried out in the manner previously described (Kipping, *loc. cit.*, 1907, 91, 217).

small quantity of residue, which is doubtless benzylethylsilicone, as it does not distil below 300° . The formation of this compound in relatively small quantities is observed in all those experiments in which samples of the chloride boiling at $196\text{--}202^{\circ}$ are employed; its presence, however, is of little importance, as, owing to its high boiling point, it is easily separated from the silicol and from the oxide. The relative proportions of benzylethylisobutylsilicol and benzylethylisobutylsilicyl oxide obtained under the above conditions are about 5 to 1 respectively.

Benzylethylisobutylsilicol, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\cdot\text{OH}$.

This compound may be obtained in a pure state by fractionally distilling the crude product collected from $150\text{--}180^{\circ}$ (see above); a large proportion of this liquid is thus obtained, boiling at $162\text{--}166^{\circ}$ 25 mm.:

0.4028 gave 0.105 SiO_2 . Si = 12.2.

0.2563 „ 0.6569 CO_2 and 0.2317 H_2O . C = 69.9; H = 10.0.

$\text{C}_{13}\text{H}_{22}\text{OSi}$ requires Si = 12.4; C = 70.1; H = 9.9 per cent.

Benzylethylisobutylsilicol is a colourless, oily liquid, having a specific gravity less than that of water, in which it is practically insoluble; it has a faint, agreeable odour. On one occasion, it was observed that a sample of the silicol, after some days, had undergone decomposition, drops of water being formed in the liquid; other samples, however, did not show this behaviour, and seemed to be stable at the ordinary temperature, so that possibly the conversion of the silicol into the oxide is brought about by traces of impurity, such as hydrochloric acid.

Benzylethylisobutylsilicyl Oxide, $\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)\cdot\text{O}\cdot\text{Si}(\text{C}_7\text{H}_7)\text{Et}(\text{C}_4\text{H}_9)$.

This liquid is obtained from the crude fraction collected between 240° and 280° (see above) by distilling it from a short-necked flask under a pressure of 20 mm.; a very large portion then passes over at $250\text{--}252^{\circ}$:

0.3507 gave 0.099 SiO_2 . Si = 13.3.

0.1832 „ 0.4918 CO_2 and 0.1628 H_2O . C = 73.2; H = 9.8.

$\text{C}_{26}\text{H}_{42}\text{OSi}_2$ requires Si = 13.3; C = 73.1; H = 9.8 per cent.

Benzylethylisobutylsilicyl oxide is a colourless, rather viscous liquid, having a specific gravity greater than that of water; it has a faint aromatic odour, and is practically insoluble in water, but is miscible with alcohol, ether, and many other organic solvents.

Sulphonation of Benzylethylisobutylsilicol with Sulphuric Acid.

Knowing that benzylethylpropylsilicol is easily converted into a sulphonic acid (which is derived from benzylethylpropylsilicyl oxide) and having all the details of the preparation and isolation of this acid

our disposal, we did not expect to meet with any great difficulty in the preparation of a sulphonic derivative of the homologous silicol just described. It was soon found, however, that, although the isobutyl compound was very easily sulphonated, or converted into products which were soluble in water, the reaction seemed to be a very complex one, and it was only after several unsuccessful attempts that we were able to isolate small quantities of the desired sulphonic acid in the form of its *l*-menthylamine salt. Further investigation showed that the nature of the product depended largely on the temperature at which the reaction was carried out.

When the silicol was well shaken with twice its volume of concentrated sulphuric acid and the mixture rapidly heated to 80°, a vigorous effervescence took place, owing to the formation of sulphur dioxide, and sulphonation was complete in less than two minutes. The product was immediately cooled, poured into water, and the clear solution neutralised with ammonia. The *l*-menthylamine salt, precipitated from this solution as an oil on adding excess of *l*-menthylamine hydrochloride, showed no tendency to crystallise, and was very readily soluble in cold moist ethyl acetate.

When the silicol was shaken with twice its volume of concentrated sulphuric acid at atmospheric temperature, it finally gave a homogeneous fluid, which, however, became turbid on standing. The *l*-menthylamine salt obtained from this product was oily, and was practically insoluble in cold ethyl acetate, a fact which showed that it was a very different substance from that obtained by sulphonating at 80°.

When the silicol was treated with a mixture of sulphuric and acetic acids, the temperature being slowly raised to 100°, an odour recalling that of isobutyl acetate was observed, but sulphonation did not take place, or was very incomplete. These and many other experiments failed to reveal a satisfactory method of sulphonation, but the following seemed to give the best results.

The silicol, in quantities of 5 grams at a time, is placed in a flask, about one and a-half times its volume of concentrated sulphuric acid is added; on shaking vigorously, the temperature rises spontaneously to about 40°, and the mixture is then rapidly heated in a bath of fusible metal to about 60–65°. Sulphonation is complete within less than two minutes, and only a slight evolution of sulphur dioxide is observed. The viscid, pale yellow product is immediately added into a large volume of water, and the acid solution neutralised with ammonia.

When this solution of the ammonium salt is directly treated with menthylamine hydrochloride, a mixture of menthylamine salts is precipitated as an oil, which may solidify immediately or only after some time; from this precipitate, the pure menthylamine salt described above can then be isolated, but generally only after a very protracted

series of fractional crystallisations, first from aqueous acetone and then from moist ethyl acetate. We therefore found it advantageous first to purify the ammonium salt of the sulphonic acid; for this purpose, the solution is evaporated, the ammonium sulphate removed by the process previously described (Trans., 1907, 91, 225), and the syrupy residue dissolved in a small quantity of methyl alcohol; ammonium sulphobenzylethylisobutylsilicyl oxide is then precipitated as a buttery mass on the addition of ethyl acetate. Even after several treatments of this kind, the salt remained in a pasty condition, and in this respect it differed widely from the well-defined, crystalline ammonium salt of sulphobenzylethylpropylsilicyl oxide.

l-Menthylamine Sulphobenzylethylisobutylsilicyl Oxide,
 $\text{SO}_3\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot(\text{C}_4\text{H}_9)\text{EtSi}\cdot\text{O}\cdot\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}\cdot 2\text{C}_{10}\text{H}_{17}\text{N}$

On adding a small quantity of an aqueous solution of *l*-menthylamine hydrochloride to that of the purified ammonium salt, an oily precipitate is obtained at first; this redissolves on stirring, but on continuing the addition of the menthylamine salt there is again produced an oily precipitate, which gradually becomes more pasty and finally granular. This product is collected, washed with water, and roughly dried. It is then recrystallised from hot moist ethyl acetate; after one or two operations, it becomes much more sparingly soluble, and is finally obtained in a pure state in flat, lustrous plates. These crystals contain water of crystallisation, which is expelled at 100° . The anhydrous salt begins to soften at 240° , and finally melts at 245° ; it is practically insoluble in anhydrous acetone and anhydrous ethyl acetate; when, however, these liquids contain water, the salt dissolves readily.

An estimation of water of crystallisation was carried out with a sample of the air-dried salt:

0.2237 lost 0.0176 H_2O . $\text{H}_2\text{O} = 7.8$.

$\text{C}_{40}\text{H}_{84}\text{O}_7\text{N}_2\text{S}_2\text{Si}_2\cdot 4\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 7.4$ per cent.

An analysis of the anhydrous salt gave the following result:

0.1489 gave 0.3346 CO_2 and 0.1293 H_2O . $\text{C} = 61.2$; $\text{H} = 9.6$.

$\text{C}_{40}\text{H}_{84}\text{O}_7\text{N}_2\text{S}_2\text{Si}_2$ requires $\text{C} = 61.5$; $\text{H} = 9.4$ per cent.

The equivalent of the salt was determined by boiling a weighed quantity of the anhydrous substance with a known volume of $N/50$ sodium carbonate until all the menthylamine was expelled, and then titrating the excess of sodium carbonate by means of $N/50$ sulphuric acid, using litmus as indicator. This method gave the value 448, the calculated equivalent of the salt of the above composition being 448. These results seem to show that the menthylamine salt is derived from the sulphonic acid of the oxide, and not from that of the silicic acid. This conclusion is confirmed by the results of molecular weight

determinations, which were made by the ebullioscopic method, using anhydrous salt and pure methyl alcohol as solvent.

Substance.	Solvent.	E.	M.W.
0.817 gram	15.9 grams	0.10	431
1.51 "	7.4 "	0.385	445

The molecular weight of the menthylamine salt of a sulphonic acid derived from the silicic acid would be 457, and that of the salt of a sulphonic derivative of the oxide would be 896; the above values therefore are such as might be expected in the case of the derivative of the oxide, as the salt is doubtless highly ionised in methyl-alcoholic solution. More conclusive results were obtained by using the sodium salt (see below).

The specific rotation of the anhydrous menthylamine salt was determined in methyl-alcoholic solution:

0.447, made up to 20 c.c., gave $\alpha = 0.65$ in a 2-dm. tube; whence $[\alpha]_D = 14.7^\circ$.

Sodium Sulphobenzylethylisobutylsilicyl Oxide.—The pure *l*-menthylamine salt is decomposed with a slight excess of sodium carbonate, and the base expelled by distilling in a current of steam, a little ethyl alcohol being added to the contents of the flask in order to prevent frothing. The residual liquid is then neutralised with acetic acid, vaporated to a small bulk, and anhydrous methyl alcohol added. This causes the precipitation of the sodium salt in colourless crystals, which are then drained and allowed to dry in the air. Molecular weight determinations were made with a sample of the salt dried at 60° , the cryoscopic method being employed with water as the solvent; the following results were employed:

Substance.	Solvent.	E.	M.W.
0.353 gram	17.3 grams	0.085	446
0.55 "	16.5 "	0.125	495

As the calculated molecular weight of the sodium salt of the sulphonic acid derived from the silicic acid is 324, and of that derived from the oxide 630, the above results fully confirm the conclusion that the sulphonic acid is derived from the oxide.

The ammonium salt of sulphobenzylethylisobutylsilicyl oxide, which has been referred to (p. 2010), is soluble in water and alcohol, but could not be obtained in a crystalline form.

The barium salt is obtained as a flocculent precipitate on the addition of barium chloride to an aqueous solution of the ammonium salt. It is insoluble in water or alcohol, but dissolves readily in a mixture of the two, and crystallises from the hot solution in glistening needles.

Sulphonation of Benzylethylisobutylsilicyl Oxide.

When benzylethylisobutylsilicyl oxide is mixed with about one and a half times its volume of concentrated sulphuric acid, the temperature

risks spontaneously to about 30°, and on warming slight darkening takes place and a little sulphur dioxide is evolved; when the temperature reaches 70°, sulphonation is complete, and the liquid is then poured into water, whereupon a clear solution is obtained if sufficient water is used. The acid solution is neutralised with ammonia and the *l*-menthylamine salt is then precipitated directly, since fractional precipitation of the ammonium salt from its solution in methyl alcohol does not seem to be of any use. The menthylamine salt obtained in this way is pasty and does not become hard even on standing. After having been washed with water, it is dissolved in aqueous acetone, from which, after some time, an oil is deposited and then crystals begin to form; on decanting the mother liquors at this stage, they subsequently deposit a crystalline product, which is collected and repeatedly crystallised from moist ethyl acetate. After many operations, a small quantity of pure *l*-menthylamine sulpho. benzylethylisobutylsilicyl oxide is thus obtained. The identity of this salt with that prepared by sulphonating the silicol is established by its melting point and by the fact that a mixture of the two salts melts at the same temperature as its components.

This method of preparing the menthylamine salt is of little practical value on account of the very small yield; however, it is of theoretical interest in that it confirms the conclusion that the menthylamine salt is derived from the oxide and not from the silicol.

The oxide was also sulphonated with chlorosulphonic acid in chloroform solution, but the results were similar to those obtained with sulphuric acid, that is to say, only a small proportion of the desired sulphonic acid seemed to be present in the product, and its isolation in the form of its *l*-menthylamine salt was an exceedingly troublesome task.

Sulphonation of Benzylethylisobutylsilicyl Chloride with Chlorosulphonic Acid.

As the sulphonation of benzylethylisobutylsilicol and of the corresponding oxide gave such disappointing results, it seemed possible that the sulphonation of benzylethylisobutylsilicyl chloride and the subsequent decomposition of the product with water might prove to be a better method for the preparation of sulphobenzylethylisobutylsilicyl oxide. Experiments showed that the chloride could be easily sulphonated with the aid of chlorosulphonic acid, and that the product was a mixture having much the same character as those obtained from the silicol and from the oxide; as, however, the direct treatment of the chloride was the most economical method as regards both time and material, most of the sulphobenzylethylisobutylsilicyl oxide required for this investigation was prepared and isolated in the following manner.

Benzylethylisobutylsiliclyl chloride is dissolved in about five times its volume of dry chloroform, the solution is cooled in ice, and the theoretical quantity of chlorosulphonic acid, also dissolved in dry chloroform, is then run in drop by drop from a tap funnel. When all the acid has been added, the product is poured on to ice, and the chloroform is removed by means of a current of steam; the residual liquid, which is sometimes milky, owing to the presence of oil, is neutralised with ammonia and allowed to stand until the oily impurity has settled. The clear liquid is then decanted, the *l*-menthylamine salt precipitated directly, and separated by filtration.

The salt thus obtained is generally solid, but somewhat pasty, and its properties seemed to vary very considerably even when portions of the same sample of chloride were sulphonated under as nearly as possible the same conditions. The crude salt is very readily soluble in cold moist ethyl acetate; when, however, it is warmed for some hours with concentrated hydrochloric acid (p. 2014), and the acid then completely removed by evaporating to dryness, the salt becomes quite hard and much more sparingly soluble in ethyl acetate. It is best purified by first crystallising it from aqueous acetone, as in this way a sparingly soluble oily by-product is first deposited; the mother liquors then give menthylamine salt in colourless, somewhat flocculent masses, which are collected, washed with water, and drained. This salt is now recrystallised from moist ethyl acetate; at first the crystals are deposited as an opaque form, owing to the presence of some impurity, but after many recrystallisations they become transparent and the pure salt ultimately obtained is identical with that prepared from the siliccol. Although this method of preparation gives a rather poor yield of pure salt, it seems, on the whole, to be the best of those which were investigated.

Simple as the sulphonation processes described above may at first sight appear, there is no doubt that in all cases the product is a mixture of a number of sulphonic acids; this is by no means surprising in view of the possibility of the formation of structural and also of optical isomerides. Even assuming that the siliccol and the chloride are completely converted into the oxide previous to, during, or after sulphonation, and that the derivatives of the oxide do not undergo hydrolysis to derivatives of the siliccol, there is still the possibility that the oxide may give rise to a mixture of ortho- and para-compounds, and each of these structural isomerides may be present in optically isomeric forms corresponding stereochemically with racemic and mesotartaric acids. In view of these considerations, objection might, of course, be taken to the employment of an optically active base (*l*-menthylamine) for the isolation of the *dl*-sulphonic acid, since its use might increase the number of the components of the mixture; we can only state, however, that we were unable to discover any better method.

The assumption just made for the sake of simplicity, that the silicol and the chloride are entirely converted into the oxide previous to, during, or after sulphonation, cannot be supported by direct evidence, as up to the present very few experiments have been carried out in order to establish the conditions under which the silicols pass into oxides and vice versa (compare Kipping, *Trans.*, 1908, 93, 461). It seemed not impossible, therefore, that the presence of a sulphonic derivative of the silicol might be one reason why the isolation of the sulphonic derivative of the oxide proved to be so troublesome. If this were the case, and the former could be converted into the latter, the nature of the product would be simplified. Guided by these considerations, we tried the effect of heating the crude *l*-menthylamine salt (prepared directly from the product of the sulphonation of the chloride) with concentrated hydrochloric acid, and the results were certainly satisfactory; after this treatment, as already stated, the menthylamine salt, which originally was very pasty and separated from aqueous acetone as an oil, crystallised from this solvent at the ordinary temperature.

By-product formed during Sulphonation.—One reason why the sulphonation of benzylethylisobutylsiliclyl chloride does not give very good results is because some of the compound is decomposed at some stage in the process, with separation of the isobutyl group; this statement is based on the following evidence.

The aqueous filtrate from the original precipitate of crude *l*-menthylamine salt (p. 2013) gives, after having been concentrated, a crystalline compound soluble in hot water, which, after repeated recrystallisation, is obtained in lustrous needles, melting at 187°. This substance is a menthylamine salt of a sulphonic acid; at first we thought it might be the salt of *p*-toluenesulphonic acid, and that it had been formed from the sulphobenzylethylisobutylsiliclyl oxide, just as this acid is known to be produced from sulphobenzylethylpropylsiliclyl oxide when the latter is hydrolysed with concentrated alkalis (*Trans.*, 1908, 93, 472). As, however, the melting point of the compound did not change on recrystallisation, and the menthylamine salt of the para-acid melts at 202°, it seemed probable that the by-product was a derivative of *o*-toluenesulphonic acid.

The equivalent of the salt, determined by titration, was found to be 310, and an analysis gave the following result:

0.1677 gave 0.3387 CO₂ and 0.1509 H₂O. C = 55.0; H = 10.0 per cent.

These values agree with those calculated for the menthylamine salt of an isobutylsulphuric acid, C₄H₉·SO₄H, C₁₀H₂₁N, which requires equivalent = 309 and C = 54.4, H = 10.0 per cent.; as, however, the compound did not yield sodium sulphate when it was boiled with sodium hydroxide, it could scarcely be an alkylsulphuric acid, and is therefore probably

salt of a sulphonic acid of the constitution $\text{CMe}_2(\text{OH})\cdot\text{CH}_2\cdot\text{SO}_3\text{H}$ or $\text{CMe}_2(\text{SO}_3\text{H})\cdot\text{CH}_2\cdot\text{OH}$. Further experiments confirmed this view; when the sodium salt, prepared from the menthylamine salt, was strongly heated with soda-lime, it yielded an inflammable liquid having an odour recalling that of a higher fatty alcohol, and this, on oxidation with chromic acid, gave a product having the odour of isobutyric acid.

These facts seem to prove that this by-product is one of the sulphonic derivatives of isobutyl alcohol, and therefore that the isobutyl group is separated from the silicon atom in the sulphonation process described above. Although it has been shown that a phenyl group combined with silicon is often easily eliminated in the form of benzene (Trans., 1907, 91, 223), this is the first case in which such a separation of an alkyl radicle has been observed. We have not made any further experiments to settle the constitution of this by-product or to ascertain exactly how it is produced; it was only examined in the hope that a knowledge of its nature would enable us to modify the conditions of sulphonation as to obtain a better yield in the preparation of sulphobenzylethylisobutylsilicyle oxide.

d-Bornylamine dl-Sulphobenzylethylisobutylsilicyle Oxide,
 $[\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}, \text{C}_{10}\text{H}_{19}\text{N}]_2\text{O}.$

On treating the pure sodium salt of sulphobenzylethylisobutylsilicyle oxide with *d*-bornylamine hydrochloride, the same phenomena are observed as in the preparation of the menthylamine salt, but the oily product solidifies only slowly. It is washed with water and crystallised from cold aqueous methyl alcohol, from which it is deposited in silky needles melting at 207–209°. It is sparingly soluble in anhydrous acetone or ethyl acetate, but dissolves freely in the presence of water; on heating its aqueous solution, hydrolysis occurs, the base being liberated.

Cinchonidine dl-Sulphobenzylethylisobutylsilicyle Oxide.

When a solution of cinchonidine hydrochloride is gradually added to an aqueous solution of the sodium salt, the oil which is first precipitated redissolves on stirring, but ultimately a sticky, silky mass is deposited. This salt is washed with water, dried, and crystallised from hot acetone containing a little methyl alcohol; it is thus obtained in small crystals. Its specific rotation was determined in methyl-alcoholic solution, using a sample of salt dried at 100°:

0.3757, made up to 20 c.c., gave $\alpha = 2.61^\circ$ in a 2-dm. tube; whence $[\alpha]_D = -69.3^\circ$.

The salt is practically insoluble in water and in anhydrous acetone, but it dissolves freely in methyl alcohol or aqueous acetone, being

deposited again as an oil on dilution with water or on evaporation. When heated fairly quickly, it softens at 175° and melts completely at 177°, darkening slightly.

Cinchonidine Hydrogen dl-Sulphobenzylethylisobutylsilicyl Oxide.

This salt is obtained by treating the normal salt with hydrochloric acid in methyl-alcoholic solution, and then evaporating the alcohol and washing the viscous residue with water; it separates from hot acetone containing a little methyl alcohol in the form of a microcrystalline powder, which deliquesces in moist air when not free from the solvent. It melts at 225–229°, and is nearly insoluble in anhydrous acetone or ethyl acetate, but dissolves much more freely if moisture be present.

Dr. R. E. Rose took part in the earlier stages of this investigation, and before he left to take up another appointment preliminary experiments had been made on the sulphonation of benzylethylisobutylsilicol and of the corresponding oxide; our cordial thanks are due to him for this assistance.

We also desire to express our indebtedness to the Government Grant Committee of the Royal Society for a grant in aid of this research.

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CCl.—*The Coumarin Condensation.*

By ARTHUR CLAYTON.

IN 1883 and 1884 von Pechmann (*Ber.*, 1883, 16, 2119; *Ber.*, 1884, 17, 929, 1646) and others found that phenols condense with ethyl acetoacetate and malic acid in the presence of concentrated sulphuric acid to form members of the coumarin series. Different phenols were found to condense with varying degrees of readiness; thus phenol, *o*- and *p*-cresols, quinol, and thymol gave extremely poor yields (phenol furnished not more than 3 per cent. of the theoretical quantity), but resorcinol, orcinol, and pyrogallol condensed very readily. Later, Pechmann (*Ber.*, 1899, 32, 3681) showed that dimethyl-*m*-aminophenol condensed readily with ethyl acetoacetate, whilst the corresponding ortho- and para-compounds yielded only traces of coumarins. Still more recently Fries and Klostermann (*Ber.*, 1906, 39, 871) condensed *m*-cresol with malic acid and ethyl acetoacetate, and showed that a plentiful yield was obtained in each case.

From the above, it is readily seen that meta-substituted phenols condense readily with ethyl acetoacetate and malic acid, and that, on the other hand, the corresponding ortho- and para-compounds are comparatively inert in this respect.

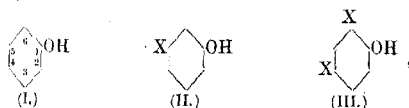
The influence of the nature of the substituent was indicated by the experiments of Pechmann (*Ber.*, 1899, **32**, 3681), in which he showed that the negative groups NO_2 and CO_2H prevented condensation.

The experiments described in the present communication were undertaken with the view of obtaining further knowledge of the influence of the position and nature of the substituent group or atom.

In order to examine the influence of substituent groups which are known to favour this condensation, 1:2:4-xyleneol, 1:3:4-xyleneol, 1:3:5-xyleneol, and 1:4:5-xyleneol were treated with a molecular proportion of ethyl acetoacetate or malic acid in the presence of concentrated sulphuric acid. 1:2:4-Xyleneol was found to yield about 50 per cent., 1:3:4-xyleneol 50 per cent., and 1:3:5-xyleneol 40 per cent of the theoretical quantity of the corresponding coumarin, whilst 1:4:5-xyleneol only condensed with extreme difficulty, not more than 1 per cent. of the theoretical yield being obtained.

The substituents which appear to exert this peculiar influence are the hydroxyl, dimethylamino-, and alkyl groups.

These results, taken in conjunction with those obtained by von Pechmann and his collaborators, show that the condensation of phenols with ethyl acetoacetate or malic acid is facilitated by the presence of one of the foregoing groups (hydroxyl, dimethylamino-, and alkyl) in position 5 (formula I), position 2 being occupied by the carbon atom taking part in the condensation. It is also seen that,



although the presence of the group in the position 4 or 6 does not render phenol more reactive, the introduction of methyl or hydroxyl in both of these positions has the same effect as that produced by substituting the hydrogen atom in position 5.

It would appear that there is some important connexion between the carbon atom taking part in the condensation and that in the para-position to this atom, for only if position 5 is masked, either by direct substitution (formula II) or by a substituent on each side (formula III), does condensation with ethyl acetoacetate readily take place.

In order to study the influence exerted by other groups, the nitrophenols, chlorophenols, cyanophenols, and the esters of the three hydroxybenzoic acid were examined.

The results obtained with the three nitrophenols confirmed those of von Pechmann (*Ber.*, 1899, 32, 3681), no condensation products being obtained.

m- and *p*-Chlorophenols were found to yield respectively 3 and 6 per cent. of the theoretical quantity of a chlorocoumarin. A coumarin derivative was not isolated in the case of *o*-chlorophenol.

The esters of the three hydroxybenzoic acids were examined in order to ascertain whether the disappearance of the acidity of the group would alter the inhibiting effect noticed by von Pechmann (*Ber.*, 1899, 32, 3681), but since the formation of coumarins does not take place, it would appear that the acidic nature of the group is not the essential condition which prevents condensation.

The cyanophenols were found to be converted into the corresponding amides of the hydroxybenzoic acids under the conditions of the experiments. In the case of *o*-cyanophenol, this conversion took place with great ease, about 90 per cent. of the theoretical quantity of salicylamide being obtained.

ψ -Cumenol was also condensed with ethyl acetoacetate and malic acid, but owing to the difficulty of isolating the products, the yields obtained could not be taken as significant. From the results obtained with 1:3:5-xylene and ψ -cumenol, a substituent in position 3 appears to inhibit condensation to some extent.

General Conclusions.

I. Phenols of the types shown in formulæ I and II (where X is an alkyl, hydroxyl, or dialkylamino-group) condense readily with malic acid and ethyl acetoacetate to form derivatives of coumarin.

II. Chlorine as a substituent has an effect similar to that of the above groups, but its influence is very much less marked.

III. The introduction of such substituents as NO_2 , CO_2H , and CO_2Et prevents condensation.

EXPERIMENTAL.

Coumarins derived from 1:2:4-Xylenol.

(i) 6:7-Dimethylcoumarin was obtained by heating a mixture of 2.5 grams of 1:2:4-xylene and 2.7 grams of malic acid with 6 c.c. of concentrated sulphuric acid until the evolution of carbon monoxide ceased. When cold, the liquid was poured on to crushed ice, and yielded a solid product, which, when crystallised from alcohol, formed white needles, melting at 148–149°:

0.1309 gave 0.3638 CO_2 and 0.0676 H_2O . C = 75.80; H = 5.74.

$\text{C}_{11}\text{H}_{10}\text{O}_2$ requires C = 75.86; H = 5.75 per cent.

(ii) 4:6:7-Trimethylcoumarin was prepared by mixing 2 grams of

1:2:4-xyleneol with an equal weight of ethyl acetoacetate, and adding a.c. of concentrated sulphuric acid. After twenty hours, the liquid as poured on to ice, and yielded a solid which crystallised from alcohol in white needles melting at $169-170^{\circ}$, the yield being 1.8 grams of the pure product:

0.1815 gave 0.5064 CO_2 and 0.1048 H_2O . $\text{C} = 76.08$; $\text{H} = 6.41$.

$\text{C}_{12}\text{H}_{12}\text{O}_2$ requires $\text{C} = 76.60$; $\text{H} = 6.38$ per cent.

(iii) 3:4:6:7-Tetramethylcoumarin.—This compound was obtained by mixing 2.5 grams of 1:2:4-xyleneol with 2.7 grams of ethyl methylacetoacetate and treating the mixture with cold concentrated sulphuric acid. 2.4 Grams of the crude product were obtained, which, on crystallisation from alcohol, yielded 1.9 grams of white needles melting at $134-135^{\circ}$:

0.1661 gave 0.4679 CO_2 and 0.1041 H_2O . $\text{C} = 76.83$; $\text{H} = 6.97$.

$\text{C}_{13}\text{H}_{14}\text{O}_2$ requires $\text{C} = 77.23$; $\text{H} = 6.93$ per cent.

Coumarins derived from 1:3:4-Xyleneol.

6:8-Dimethylcoumarin, the condensation product of 1:3:4-xyleneol and malic acid, was prepared in the usual manner. These substances were heated with concentrated sulphuric acid, and the cold mixture poured on to crushed ice, when a black tar was deposited, which, after stirring for some time, became solid. This was extracted with petroleum, and the solution yielded well-defined, white needles, melting at 95° . About 30 per cent. of the theoretical yield was obtained, but the difficulty of isolating the product probably caused a considerable loss of material:

0.1692 gave 0.4689 CO_2 and 0.0888 H_2O . $\text{C} = 75.56$; $\text{H} = 5.84$.

$\text{C}_{11}\text{H}_{10}\text{O}_2$ requires $\text{C} = 75.86$; $\text{H} = 5.75$ per cent.

4:6:8-Trimethylcoumarin was obtained by condensing equal weights of ethyl acetoacetate and 1:3:4-xyleneol in the presence of cold concentrated sulphuric acid. The solution was allowed to remain for two days, and was then poured on to crushed ice. The resulting solid crystallised from alcohol in white needles, melting at $116-117^{\circ}$. The yield was about 50 per cent. of the theoretical amount:

0.1808 gave 0.5070 CO_2 and 0.1079 H_2O . $\text{C} = 76.47$; $\text{H} = 6.63$.

$\text{C}_{12}\text{H}_{12}\text{O}_2$ requires $\text{C} = 76.60$; $\text{H} = 6.38$ per cent.

3:4:6:8-Tetramethylcoumarin.—This compound resulted from the condensation of 1:3:4-xyleneol and ethyl methylacetoacetate in molecular proportion. The product was obtained without difficulty, and crystallised from alcohol in white needles melting at $110-111^{\circ}$. The yield was about 25 per cent. of the theoretical amount:

0.1332 gave 0.3754 CO_2 and 0.0832 H_2O . $\text{C} = 76.85$; $\text{H} = 6.94$.

$\text{C}_{13}\text{H}_{14}\text{O}_2$ requires $\text{C} = 77.23$; $\text{H} = 6.93$ per cent.

Coumarins derived from 1:3:5-Xylenol.

4:5:7-*Trimethylcoumarin*.—1:3:5-Xylenol and ethyl acetoacetate were condensed together in the usual manner. The crude product crystallised from alcohol in brilliant flakes, having a pearly lustre and melting at 175—176°. From 35 to 40 per cent. of the theoretical quantity was obtained:

0.0964 gave 0.2690 CO₂ and 0.0562 H₂O. C = 76.10; H = 6.48.

C₁₂H₁₂O₂ requires C = 76.60; H = 6.38 per cent.

3:4:5:7-*Tetramethylcoumarin* was prepared from ethyl methylacetoacetate and 1:3:5-xylenol in the presence of cold concentrated sulphuric acid. A pink product was obtained, but on repeated crystallisation from alcohol this colour grew fainter, and finally disappeared. One gram of 1:3:5-xylenol yielded 0.15 gram of crystals melting at 154°:

0.0667 gave 0.1880 CO₂ and 0.0427 H₂O. C = 76.92; H = 7.11.

C₁₃H₁₄O₂ requires C = 77.23; H = 6.93 per cent.

Coumarin derived from 1:4:5-Xylenol.

5:8-*Dimethylcoumarin*.—Twelve grams of 1:4:5-xylenol and 13 grams of malic acid were ground together and treated with 30 c.c. of concentrated sulphuric acid, the mixture being heated until the evolution of carbon monoxide ceased. After twenty hours, the liquid was poured on to crushed ice. The tar which separated did not solidify for several days, but, on dissolving it in a 15 per cent. solution of sodium hydroxide and subsequently acidifying with hydrochloric acid, a light brown powder was obtained. This was very impure, and was therefore extracted with petroleum, the extract yielding about 0.2 gram of fine, white needles, melting at 122—123°:

0.0542 gave 0.1504 CO₂ and 0.0296 H₂O. C = 75.59; H = 6.03.

C₁₁H₁₀O₂ requires C = 75.86; H = 5.75 per cent.

Coumarins derived from ψ -Cumenol.

5:6:8-*Trimethylcoumarin*.—Three grams of ψ -cumenol and 3 grams of malic acid were heated with 8 c.c. of concentrated sulphuric acid in the usual manner. After crystallising the product from alcohol, about 40 per cent. of the theoretical yield of crystals was obtained. When pure, the crystals melted at 114—115°:

0.1890 gave 0.5302 CO₂ and 0.1087 H₂O. C = 76.50; H = 6.39.

C₁₂H₁₂O₂ requires C = 76.60; H = 6.38 per cent.

4:5:6:8-*Tetramethylcoumarin* was obtained by the condensation of ψ -cumenol and ethyl acetoacetate, but there appeared to be a side

reaction taking place, the mixture frothing continually. After twenty hours, the solution was poured on to crushed ice, and yielded a quantity of an oily product, which set into an almost solid mass after forty-eight hours. The substance so obtained was extracted with benzene, and crystallised from the same medium. Subsequent crystallisation from alcohol yielded pure white needles, melting at $14-115^{\circ}$. About 12 per cent. of the theoretical yield was obtained:

0.0775 gave 0.2200 CO_2 and 0.0485 H_2O . $\text{C} = 77.42$; $\text{H} = 6.82$.

$\text{C}_{13}\text{H}_{14}\text{O}_2$ requires $\text{C} = 77.23$; $\text{H} = 6.93$ per cent.

3:4:5:6:8-Pentamethylcoumarin.—This coumarin resulted from the condensation of equal weights of ethyl methylacetoacetate and coumaenol. The solution of these two substances in concentrated sulphuric acid was allowed to remain for twenty hours, and was then poured on to crushed ice. The oil which separated could not be made to solidify after two weeks, but by cooling it to -15° it was obtained sufficiently viscous for removal from the aqueous liquid. The product resisted all attempts at crystallisation, but it was found that by rubbing it with a few drops of ether, a white, perfectly solid substance was obtained. This was immediately strained on a porous tile, and then crystallised from petroleum. The white needles obtained in this manner melted at 127° . The yield was very small, owing to loss during the isolation of the compound:

0.1036 gave 0.4387 CO_2 and 0.1036 H_2O . $\text{C} = 77.37$; $\text{H} = 7.44$.

$\text{C}_{14}\text{H}_{16}\text{O}_2$ requires $\text{C} = 77.78$; $\text{H} = 7.41$ per cent.

Coumarins derived from m-Chlorophenol.

7-Chlorocoumarin was obtained by treating a mixture of 5 grams of *m*-chlorophenol and 5 grams of malic acid with 13 c.c. of concentrated sulphuric acid. The liquid was heated until the evolution of carbon monoxide ceased, and was then allowed to cool. After two hours, the acid solution was poured on to crushed ice in order to precipitate the chlorocoumarin. The semi-solid product was extracted three times with petroleum, and, on evaporating the ether, white needles (0.3 gram) were obtained, which, after further crystallisation from alcohol, melted at 129° :

0.0607 gave 0.0481 AgCl . $\text{Cl} = 19.60$.

0.0856 „ 0.1873 CO_2 and 0.0208 H_2O . $\text{C} = 59.67$; $\text{H} = 2.57$.

$\text{C}_9\text{H}_5\text{O}_2\text{Cl}$ requires $\text{Cl} = 19.67$; $\text{C} = 59.83$; $\text{H} = 2.77$ per cent.

7-Chloro-4-methylcoumarin was obtained by condensing 5 grams of *m*-chlorophenol with 5 grams of ethyl acetoacetate in the presence of 1 c.c. of cold concentrated sulphuric acid. After twenty hours, the

liquid was poured into ice-water, and the solid product separated by filtration. Crystallisation from alcohol yielded 0.45 gram of white needles, or about 6 per cent. of the theoretical amount. The substance melted at 144° :

0.0956 gave 0.0747 AgCl. Cl = 18.38.

0.1073 „ 0.2427 CO_2 and 0.0365 H_2O . C = 61.69; H = 3.78.

$\text{C}_{10}\text{H}_7\text{O}_2\text{Cl}$ requires Cl = 18.25; C = 61.70; H = 3.60 per cent.

Coumarins derived from p-Chlorophenol.

6-Chlorocoumarin resulted from the condensation of 13 grams of *p*-chlorophenol and 14 grams of malic acid in the presence of 30 c.c. of concentrated sulphuric acid, the solution being heated as usual. After cooling the liquid and pouring it into ice-water, a black tar was deposited. This product, on extraction with petroleum, furnished a white solid, which crystallised from alcohol in tiny, white needles, melting at $161\text{--}162^{\circ}$. The yield was about 2.5 per cent. of the theoretical amount.

The substance has a well-marked odour of coumarin, and is possibly identical with the “ β -chlorocoumarin” obtained by Bäsecke (*Annalen*, 1870, 154, 84), the melting point of this substance being given as 162° :

0.0686 gave 0.0547 AgCl. Cl = 19.81.

0.1048 „ 0.2316 CO_2 and 0.0285 H_2O . C = 60.26; H = 3.02.

$\text{C}_9\text{H}_5\text{O}_2\text{Cl}$ requires Cl = 19.67; C = 59.83; H = 2.77 per cent.

6-Chloro-4-methylcoumarin.—This substance was prepared by mixing 5 grams of ethyl acetoacetate, 5 grams of *p*-chlorophenol, and 12 c.c. of cold concentrated sulphuric acid, the mixture being allowed to remain for twenty hours. After that period, the liquid was poured into ice-water. The white solid which separated was crystallised from alcohol, from which it separated in white needles, melting at $184\text{--}185^{\circ}$. The yield was about 2.7 per cent. of the theoretical quantity:

0.1406 gave 0.1032 AgCl. Cl = 18.16.

0.1779 „ 0.4016 CO_2 and 0.0568 H_2O . C = 61.60; H = 3.55.

$\text{C}_{10}\text{H}_7\text{O}_2\text{Cl}$ requires Cl = 18.25. C = 61.70; H = 3.60 per cent.

Experiments with *o*-chlorophenol did not result in the isolation of any coumarin, the oils and tars which resulted proving to be uncrystallisable. The odour of these products, however, showed that condensation had taken place to some slight extent.

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617 10* or TRANS., 1888, 53," read "TRANS., 1887, 51, 610."

VOL. LXX (ABSTR., 1896).

PART I.

33 9 for " $\text{COOEt}\cdot\text{C}(\text{OH})(\text{CO}\cdot\text{NHPh})\cdot\text{C}(\text{OH})(\text{CO}\cdot\text{NHPh})\cdot\text{COOEt}$ "
read " $\text{COOEt}\cdot\text{CH}(\text{O}\cdot\text{CO}\cdot\text{NHPh})\cdot\text{CH}(\text{O}\cdot\text{CO}\cdot\text{NHPh})\cdot\text{COOEt}$."

VOL. XCII (ABSTR., 1907).

PART I.

137 1 for "2-Nitroisophthalic acid" read "2-Nitroisophthalic acid."

VOL. XCIII (TRANS., 1908).

949 3, 6 for "23°" read "21°"
949 5 "d₂₃" "d₂₁"
1798 3* " $(\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2)_2\cdot\text{H}_2\text{PtCl}_6$ " read " $(\text{C}_{20}\text{H}_{20}\text{O}_4\text{N})_2\cdot\text{H}_2\text{PtCl}_6$ "
1799 13* " $(\text{C}_{19}\text{H}_{20}\text{O}_4\text{N})_2\cdot\text{H}_2\text{PtCl}_6$ " read " $(\text{C}_{19}\text{H}_{20}\text{O}_4\text{N})_2\cdot\text{H}_2\text{PtCl}_6$ "
1899 16 " $\text{C}_{13}\text{H}_{12}\text{O}_4\text{N}_2\text{As}_2\cdot 2\frac{1}{2}\text{H}_2\text{O}$ " read " $\text{C}_{13}\text{H}_{12}\text{O}_4\text{N}_2\text{AsNa}_2\cdot 2\frac{1}{2}\text{H}_2\text{O}$."

VOL. XCIV (ABSTR., 1908).

PART I.

408 21 for "ROLEKER" read "ROLKER."
927 1, 2, 3 should read "p-Nitrobenzenearosalicic acid, m. p. 256°; the condensation is proved by its yielding aminosalicic acid and p-phenylenediamine when reduced by sodium hyposulphite in hot aqueous solution."
927 13 for "hydrogen" read "hypo."
927 21, 22 "diaminodiphenyl derivative of carbamide" read "diaminodiphenylcarbamide derivative."
927 24 before "p-aminophenol" insert "diazotised."
927 25 "aminosalicic acid" insert "diazotised."

* From bottom.

Studies on the Viscosity and Conductivity of some Aqueous Solutions. Part I. Solutions of Sucrose, Hydrogen Chloride, and Lithium Chloride.

By W. HEBER GREEN, D.Sc.

It has been suggested by various writers that there is some condition, present undetermined, which complicates conductivity results obtained in very dilute solutions. On the other hand, few accurate measurements have been made with concentrated solutions, although these should be of great value in deciding the relation connecting the conductivity, viscosity, density, and concentration of solutions.

Part I (this paper) is a contribution in the latter direction, and describes measurements of the viscosity of sucrose solutions and the viscosity and conductivity of hydrogen chloride and lithium chloride solutions over ranges of concentrations limited only by the solubilities of each substance.

Part II (following paper) is a contribution towards the elucidation of the connexion between ionic mobility and physical fluidity in electrolytic solutions.

Viscosity of Aqueous Solutions of Sucrose (at 18° and 25°).

The sucrose used was a good sample of "brewer's crystals," specially obtained from the Australian Refining Company's works at the courtesy of their chemist, Mr. Miller, to whom I am also indebted for the following analysis :

Sucrose	99.96
Levulose	trace
Other organic matter.....	0.01
Soluble ash	0.01
Moisture	0.02
	<hr/>
	100.00

The crystals were slightly deliquescent, possibly due to the presence of potassium carbonate as an impurity on the surface of the crystals, and which it has since been discovered can be removed by washing with absolute alcohol.

The concentration of each solution was determined from the density, checked, in a few cases, by direct weighing of the sucrose in water employed.

The densities were determined by the use of a pear-shaped 25 c.c. bottle.

specific gravity bottle,* the stopper of which had been carefully re-ground with flour emery.

A series of determinations of the density of water showed that the maximum error liable to occur was 0.00002, and that the two sources of error to be guarded against were the presence of small air bubbles and variations in the buoyancy of the atmosphere, in which respects the other forms of pycnometer are in no way superior.

The observations of Balling and Brix, Gerlach, Kohlrausch and Hallwack (*Wied. Ann.*, 1894, **53**, 30), and Wade (*Trans.*, 1899, **95**, 270) on the density of sucrose solutions at or about 18° were carefully compared by plotting the functions $\frac{S-1}{N}$ and N against one another.

S denotes the specific gravity of the solution relative to water at the same temperature, and N its concentration expressed in gram-molecules per litre. A curve was then drawn through the most concordant determinations, and from this the smoothed values used throughout were calculated, and have since been found to be very concordant with the revised table given in Landolt and Börnstein's *Physikalisch-chemische Tabellen*, 1905, p. 364.

As no trustworthy data for 25° were then available, several solutions were made up, and their densities carefully observed. These results were similarly plotted, calculated, and used in this paper.

The density of the solid sucrose was determined by displacement of dry petroleum ("kerosene"), with the results: D_4^{25} 1.5871, D_4^{25} 1.5917, $N = 4.639$, $\phi = 215.6$. Previous determinations are: Schraeder, 1.588 (at 4°); Maumene, 1.5951 (at 15°); Gerlach, $D_{17.5}^{25}$ 1.58046. My value is somewhat higher than that obtained by Gerlach.

Several interesting features were noted whilst working with these solutions.

The apparent molecular volume of sucrose (ϕ) may be calculated from the formula $\phi = \frac{M}{d_w} - 1000 \frac{S-1}{N}$, where d_w = density of water, and $\frac{M}{d_w} = 342.5$ at 17.5°; and at about this temperature, ϕ may be taken as varying from 210.0 for solutions of extreme dilutions to 220.0 for a 100 per cent. solution ($= 4.55 N$).

I find that the formula $\phi = 210.00 + 1.75 N + 0.1 N^2$ affords the simplest method of expressing the densities of sucrose solutions, with an error of not more than one unit in the fourth decimal figure.

It may be noted that for a 65 per cent. solution at 25° (practically a saturated solution), ϕ is the same as for the solid crystal, namely, 215.6. Although a 65 per cent. solution is a saturated one at the

* A groundless prejudice seems to exist against the use of this very convenient and really highly-accurate instrument. See *Chem. News*, 1908, **98**, 49, for some notes on the determination of specific gravity and the regulation of thermostat.

ordinary temperature, yet, owing to the extreme viscosity of strong syrups, supersaturated solutions may be readily worked with, and, even if crystallisation be accidentally started, days and weeks may elapse before the excess of sucrose has all been precipitated, unless considerable agitation takes place. Consequently, the specific gravity of solutions containing up to 99 per cent. of sucrose has been determined (see Watt's *Dictionary*), from which we can obtain, with very little extrapolation, the density of a 100 per cent. sucrose solution. This is not identical with the density of ordinary crystalline sucrose. I can find, however, no original record of any observations on solutions containing more than 70 per cent.

Determination of Viscosity.—The apparatus used was a modification of the Poiseuille-Detwold form of tube, and was designed to admit of convenient changing of the liquid to be examined and rapid cleaning of the apparatus.

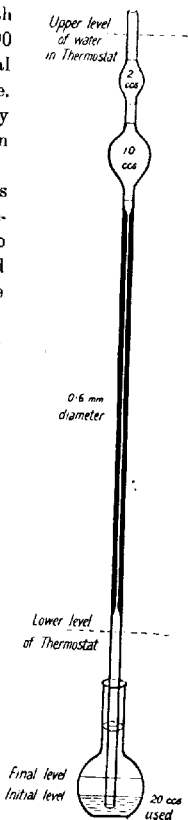
The capillary was about 0.72 mm. in diameter and 34 cms. long, whilst the capacity of the bulb from the upper to the lower mark was 9.27 c.c. The lower end of the capillary was sealed on to a piece of wider tube, which tipped underneath the surface of the liquid in a reservoir flask of about 30 c.c. capacity.

Exactly 20 c.c. of liquid were used in each experiment, and, as by means of levelling screws a graduation mark on the neck of the bulb was made to coincide with a line etched about 5 cms. from the lower end of the tube, the "working head" of liquid (nearly 51 cms.) could be adjusted with considerable accuracy to the same value for each experiment.

As the temperature-coefficient is for some liquids as high as 8 per cent. per 1° , accurate temperature regulation is essential. The viscometer was jacketed by a water-bath with glass inlets, and the temperature was regulated by an improved toluene regulator, capable, if required, of preventing fluctuations of more than 0.01° .

All temperatures were read to 0.01° from thermometers, graduated 0.1° or 0.04° , which had been certified at Kew (or had been directly compared with a Kew certificated thermometer), and the

FIG. 1.



zero points of which were re-determined from time to time. The temperatures given may be relied on to within 0.02° .

The time of outflow was measured in the usual way by a stop-watch (0.25 second), the rate of which was occasionally determined and found not to have altered sufficiently to affect any of the results.

With water at 25° , the time measured was only about 90 seconds, but the apparatus was only used to measure the viscosity of concentrated sucrose solutions, and in one case the time of outflow was nearly eight hours. This entailed a multiplication of experiments in order to bring the error below 0.1 per cent.*

Standardisation of the Viscometer.---In the sequel, the symbols used have the following significance:

η = coefficient of viscosity in C.G.S. units.

h = average head of liquid driving it through the capillary.

r = mean radius of capillary.

l = length of capillary.

v = volume of bulb.

g = gravitation constant.

d = density of liquid.

t = time of outflow.

$f = \frac{1}{\eta}$ = fluidity.

For a vertical capillary tube apparatus, the formula $\eta = \frac{\pi}{8} \cdot \frac{hgr^4}{l} \cdot dt$ assumes that the work done by the pressure (due to the head of liquid, is entirely converted into heat inside the tube, the liquid escaping without appreciable kinetic energy.

The Couette-Finkener-Wilberforce correction for any such loss of kinetic energy is given by the second term of the formula:

$$\eta = \frac{\pi}{8} \cdot \frac{hgr^4}{lv} \cdot dt - \frac{v}{8\pi l} \cdot \frac{d}{t} \\ = A \cdot dt - B \cdot \frac{d}{t},$$

where A and B are constants for any given apparatus.† This assumes

* A more convenient form of tube would have two pear-shaped bulbs, a lower one of about 10 c.c. capacity for general use, and an upper one of about 15 c.c. for use with more viscous liquids. The diameter of the capillary should be slightly decreased, say to 0.6 mm., giving, for water at 20° , a time of outflow of about four minutes (see Fig. 1).

† Knibbs (*J. Roy. Soc. New South Wales*, 1895, 29, 77; 1896, 30, 186) has very thoroughly investigated all the recorded experiments on the viscosity of water and concludes that the correct formula should be:

$$\eta = \frac{\pi}{8} \cdot \frac{hgr^4}{lv} \cdot dt - 1.12 \frac{v}{8\pi l} \cdot \frac{d}{t}$$

that is, that the correction which has been adopted by all the later workers should

that the area of cross section of the capillary and orifice is uniform throughout, but if the latter be contracted or expanded the correction will be accordingly affected.

It is apparent that if the bore of the capillary be gradually increased and made funnel-shaped at the junction with the under-tube, so that the momentum of the outflowing liquid is uniformly shared by that immediately surrounding it in the gradually widening part of the funnel and without producing eddy currents, then the kinetic energy lost outside the tube may be diminished to a negligible quantity and any correction abandoned.

Critical Examination of Published Values for the Viscosity of Water.

It was decided to calibrate the apparatus by observing the times of outflow of water at temperatures varying from 0° to 35°, and to compare them with the true viscosities as determined by previous experimenters.

Increased by 12 per cent. In the present paper, however, this amendment has not been introduced, as all the tedious calculations involved had been completed before this paper was observed, and the discrepancy involved is only about 0.1 per cent. of the total value of the viscosity.

By many experimenters, the influence of this correction appears to have been entirely neglected, and the viscosity is simply taken as proportional to "times of outflow" (Dunstan, *Trans.*, 1904, **85**, 817; Fawsitt and Lowry, *Electrochemist and Metallurgist*, 1904, **3**, 800; Traube, *Physico-chemical Methods*, p. 54; Hartley, *Transactions and Applebey, Trans.*, 1908, **93**, 543). The following quotation, from a recent session at the Faraday Society, is typical of the misunderstanding which seems to exist as to the influence of the form of the tube on this correction: "It is important that the capillary should be straight and cut off squarely at each end, and should be drawn out or blown out continuously with the bulb of the pipette. Even when consideration was taken the relative times of outflow depended on the diameter and length of the tube, but this variation could be eliminated if a correction were made and the viscosity deduced from a formula $\eta = at + bt^2$, instead of from the linear relation $\eta = at$; the constant b could be determined by calibrating the tube with a standard liquid or by determining the time of outflow of water at two temperatures." Undoubtedly, the correct method is to standardise the viscometer by using two or more liquids of known viscosity, but the formula should be $\eta = Adt + Bdt^2$, and it will be found that, when the capillary has been blown out continuously with the wider portions of tubing, the correction will tend to vanish; in other cases, it may amount to 2 or 3 per cent.

Another possible source of error that does not seem to be generally appreciated is that some of the liquid will remain on the walls of the bulb after it is apparently empty, and the amount so remaining will depend on the capillarity and viscosity of the liquid. Fortunately, it also depends on the time available for the draining of the surfaces, and so this surface skin does not cause the error it otherwise would. Some experiments were carried out to test this point, and the outflowing volumes were compared under various conditions. The results showed that the error from this source is not more than 0.1 per cent., which is about the limit of experimental error in other directions. Even this can be diminished by the use of elongated instead of spherical bulbs.

For this purpose, the accurate measurements of Thorpe and Rodger must be regarded as superseding the pioneer work on this subject, of which they give a full account and criticism (*Phil. Trans.*, 1894, A, 195, 397). Since then the absolute values for the viscosity of water have also been accurately measured by R. Hosking (*Phil. Mag.*, 1900, [v], 49, 274).

Each individual experiment was plotted in the form of a suitable complex function of viscosity and temperature, and finally a series of smoothed values obtained independent of isolated discordant observations.

Thorpe and Rodger carried out two sets of experiments on water: in the one set, observations were taken at intervals of about 9° between 0° and 100° , and in the other, they made twelve experiments between 0° and 8° in order to test the question as to any abnormality at 4° comparable with the alteration in density. Their first set agree well, with one exception at 5.4° , but the lower series are not so concordant with one another or with the first series.

Hosking's observations were taken at almost the same temperature-intervals, but do not give such consistent results as Thorpe and Rodger's first series, and, whilst the value given for η at 0° is 0.9 per cent. higher than that of Thorpe and Rodger's, his results at temperatures between 10° and 35° are about 0.6 per cent. higher than the corresponding figures given by them.

From the construction of Hosking's apparatus, the capillary could be removed and its constants measured with great accuracy at any time, whilst Thorpe and Rodger's absolute value of η depends on a weighing of about 0.013 gram of mercury, which, as the pointer of the balance moved through seven divisions with an overload of 1 milligram, may reasonably be taken as correct to about 0.03 milligram, involving an error of 0.25 per cent. in the weight of the mercury and, consequently, of 0.5 per cent. in the value of η ; besides this, there is also to be taken into account the difficulty of measuring the length of their capillary (about 4 cms.) accurately through the differing thicknesses of glass, and the uncertainty as to whether any alteration had taken place during the sealing together of the apparatus.

So, for these reasons, Thorpe and Rodger's values have been taken as relative, and, after increasing by 0.6 per cent., were then given equal weight to the results obtained by Hosking.*

* This conclusion is borne out by Knibbs (*loc. cit.*), who, in discussing Thorpe and Rodger's measurements before Hosking's work had been published, states: "For absolute values of the viscosity the data are, therefore, not satisfactory, in fact, Poiseuille's tubes are the only ones that appear, so far, to have been thoroughly measured. . . . The imperfect knowledge of the effective radius of the tube, however, in no way prejudices the relative values of the fluidity for various temperatures; it can only affect the absolute value of the viscosity constant."

The average values given in table I are consistently close to Slotte's figures (*Wied. Ann.*, 1883, 20, 262), and are probably the most correct obtainable from the data at present available (compare Hosking, *r. Roy. Soc., New South Wales*, 1908, 42, 34).

TABLE I.

Summary of Determinations of the Viscosity of Water.

	Thompson and Rodger		Hosking			Best average values,	Slotte.
	Original.	Recalculated + 0.6%.	Original.	Recalculated.	W.H.G.		
2.	0.01778	0.0178875	0.01794	0.017940	0.017930	0.017919	0.01808
5	0.015095	0.015190	0.01520	0.015215	—	0.015201	0.01524
8	—	0.013872	—	0.013883	0.013864	0.013873	—
10	0.013025	0.0130985	0.01309	0.013098	0.013116	0.013104	0.01314
15	0.011335	0.0114185	0.01143	0.011428	0.011416	0.011421	0.01144
18	—	0.010582	—	0.010588	0.010578	0.010583	—
22	0.010015	0.0100755	0.01009	0.0100775	0.010071	0.010075	0.01008
25	0.00891	0.008962	0.00897	0.008961	0.008949	0.008954	0.00896
30	0.007975	0.0080285	0.00802	0.0080285	0.008030	0.008029	0.00803
35	0.00720	0.007244	0.00724	0.0072425	0.007255	0.007247	0.00724

In order to standardise the apparatus in terms of the above values, nearly 200 observations were taken of the times of outflow of water between 8° and 36°; the time of outflow at 0° was found to be 174.45 seconds, the mean of nine readings taken with the thermostat filled with clean, freshly-fallen hail.

Plotting a suitable function, enabled the smoothed values of dt to be read off for every 5°.

Then as $\eta = A dt - B \frac{d}{t}$, where η is taken from the average values given above, and t is the observed time of outflow; if we plot:

$$\begin{cases} \frac{\eta}{dt} \text{ as "y",} \\ \frac{1}{t^2} \text{ as "x"} \end{cases}$$

the points should lie on a straight line making an angle ψ with the axis "x,"

$$\begin{aligned} \text{then } B &= \tan \psi \\ \text{and } A &= y + Bx. \end{aligned}$$

The coincidence of the experimental values with a straight line when plotted in this way (see Fig. 2) proved the applicability of the formula to the tube employed, the greatest discrepancy observed being 1.6 per cent.

The values found for A and B were:

$$A = 1.0372 \times 10^{-4}$$

$$B = 285.8 \times 10^{-4},$$

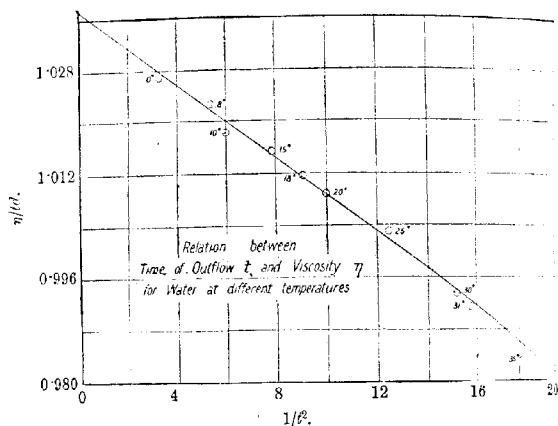
and in the work to be described the following were adopted as the

true viscosities and fluidities of pure water at the temperatures indicated:

θ .	η .	f .
17.82°	0.010630	94.07
18.00	0.010580	94.52
24.90	0.008977	111.40
25.00	0.008953	111.67

Method of Experimenting.—In each experiment the solution, of which exactly 20 c.c. were placed in the reservoir flask, was drawn up past the mark above the bulb and the stirrer started, while the temperature was kept as constant as possible for half an hour at least; then an observation was taken, which for the most concentrated sucrose solution lasted for more than 28,000 seconds (nearly eight hours), and

FIG. 2.



for the most dilute, about 100 seconds. If the time was prolonged, temperature readings were taken at regular intervals, and the average temperature calculated.

The solution was again brought into position near the upper mark, and kept there (the temperature of the bath having been meanwhile slightly altered) for at least half an hour before the next observation was taken.

Three or more readings were taken with each solution at temperatures slightly above and below the standard; this allowed of the temperature-coefficient being approximately calculated, and enabled the errors due to evaporation and change of concentration of the solution while in the viscometer to be detected and compensated for.

The density was simultaneously determined, as previously described, on another portion of the same solution in a 25 c.c. specific gravity bottle, which was allowed to take the temperature of a water-bath permanently kept within 0.04° of the standard.

The following example shows in detail how the data obtained from each solution were utilised :

Viscosity of Sucrose Solution (IV). Temp. = 25°.

(i.)		(ii.)		(iii.)	
<i>t.</i>	<i>θ.</i>	<i>t.</i>	<i>θ.</i>	<i>t.</i>	<i>θ.</i>
0'	25.16°	0'	25.40°	0'	25.00°
3	25.15	4	25.40	4	24.98
5	25.15	7	25.39	7	24.98
8	25.15	10	25.39	10	24.98
10	25.14				
Total time of outflow = 10' 23.6", <i>t.</i> = 623.6 seconds, mean <i>θ</i> = 25.15°.		Total time of outflow = 10' 17.0", <i>t.</i> = 617.0 seconds, mean <i>θ</i> = 25.395°.		Total time of outflow = 10' 27.5", <i>t.</i> = 627.5 seconds, mean <i>θ</i> = 24.985°.	

$$\begin{aligned}
 (\text{Temp.-coeff.}) &= 0.040 \\
 \therefore t_{25.0} &= 627.0 \text{ secs.} \\
 \text{and } \eta &= \frac{A(t - B)}{t} \\
 &= 0.07816 - 0.000055 \\
 &= 0.07810.
 \end{aligned}$$

The determination of the density of the solution (sp. gr. bottle, No. 27) gave $d_{25}^{25} = 1.20196$, hence $S - 1 = 0.20542$,

but from smoothed curve, $\frac{S - 1}{n} = 0.12915$,

therefore $n = 1.5905$.

The normality is obtained approximately by interpolation from the density tables, and finally by reference to the curve for the exact value of $\frac{S - 1}{n}$ at that concentration.

The concentrations of sucrose solutions are almost invariably given in reference books as percentages: for most practical purposes they could have been so expressed in this paper, and even in the case of the mixtures of sucrose and lithium chloride solutions there is no theoretical significance apparent in the ratios of their molecular concentrations. However, the calculations were simplified by expressing the concentrations in terms of "*n*," and the nomenclature was also brought more into harmony with that necessarily adopted for the electrolytic solutions examined.

EXPERIMENTAL.

Viscosity of Sucrose Solutions.

$$\eta = A\Delta t - B \frac{d}{t}.$$

η = coeff. of viscosity.

$$A = 1.0372 \times 10^{-4}$$

Constants for the

t = time of outflow in seconds. $B = 285.8 \times 10^{-4}$ apparatus.

n = normality in gram-molecules of sucrose per litre.

TABLE II. $\theta = 18.00^\circ$.

c^{25}_s	t	$A\Delta t$	Bd/t	η	n	$\log. \left\{ \frac{\log. \eta/n}{n} \right\}$
0.99865	104.75	0.010850	0.000270	0.010580	0	
1.01884	117.6	0.012428	0.000248	0.012180	0.1518	1.6023
1.01945	117.8	0.012456	0.000247	0.012209	0.1565	1.5963
1.03937	131.5	0.014500	0.000221	0.014279	0.3078	1.6250
1.04276	138.2	0.014946	0.000216	0.014730	0.3337	1.6329
1.08555	192.35	0.021657	0.000161	0.021496	0.6607	1.6678
1.1352	315.8	0.037184	0.000103	0.037081	1.0439	1.7171
1.14842	379.7	0.044154	0.000089	0.044065	1.1468	1.7323
1.1758	533.7	0.065084	0.000063	0.065021	1.3609	1.7928
1.17632	535.5	0.065344	0.000063	0.065281	1.3650	1.7924
1.18059	572.6	0.070185	0.000059	0.070076	1.4010	1.7677
1.1857	614.5	0.075570	0.000055	0.075515	1.4310	1.7754
1.23435	1423.5	0.18225	0.000025	0.18223	1.8248	1.8307
1.2794	3946	0.52363	0.000009	0.52362	2.1876	1.8890
1.3180	12481	1.7062		1.7062	2.5030	1.9454
*1.3415	28630	3.9836		3.9836	2.6273	1.9794

* This solution was supersaturated, and the sucrose slowly crystallised after the readings had been taken.

TABLE III. $\theta = 25.00^\circ$.

c^{25}_s	t	$A\Delta t$	Bd/t	η	n	$\log. \left\{ \frac{\log. \eta/n}{n} \right\}$
0.99712	89.6	0.009267	0.000318	0.008953	0	
1.01976	101.35	0.010719	0.000288	0.010431	0.1723	1.5849
1.03358	110.15	0.011809	0.000268	0.011541	0.2779	1.5983
1.04788	121.0	0.013151	0.000248	0.012903	0.3875	1.6120
1.05828	158.4	0.017797	0.000195	0.017602	0.6603	1.6479
1.06158	170.2	0.019270	0.000182	0.019088	0.7242	1.6569
1.10062	184.7	0.021084	0.000170	0.020914	0.7947	1.6661
1.12228	228.0	0.026540	0.000153	0.026387	0.9829	1.6878
1.14240	283.3	0.033568	0.000115	0.033458	1.1205	1.7083
1.17124	403.8	0.049054	0.000083	0.04897	1.3472	1.7386
1.20196	627.0	0.07816	0.000055	0.07810	1.5905	1.7719
1.23140	1040.0	0.13283	0.000030	0.13280	1.8255	1.8072
1.25964	1828.0	0.23883	0.000020	0.2388	2.0517	1.8120
1.28676	3490.0	0.4658	0.000010	0.4658	2.271	1.8782

The smoothed values given in table IV were calculated from the curves obtained by plotting a suitable function, namely, $\log. \left\{ \frac{\log. \eta/n}{n} \right\}$, of the above experimental values. The plotting of η itself (which

varies from 0.01 to about 4.0), with the necessary accuracy, was unpracticable. It will be observed that the temperature-coefficient increases considerably with the concentration.

TABLE IV.

Viscosities and Fluidities of Sucrose Solutions as Calculated by Interpolation from Smoothed Curves.

α = Average percentage temperature-coefficient between 18° and 25°.

c.	$\theta = 18^{\circ}00'$			$\theta = 25^{\circ}00'$			$\alpha = \frac{\eta_{18} - \eta_{25}}{25 - 18} \times \frac{200}{\eta_{18} + \eta_{25}}$
	d^{18}	η	f	d^{25}	η	f	
0.9	0.99565	0.010580	94.51	0.99712	0.008953	111.67	2.390
1.1	1.01288	0.011600	88.19	1.01028	0.009766	102.38	2.454
1.2	1.02509	0.012776	78.26	1.02339	0.010705	93.41	2.520
1.4	1.05119	0.015744	63.51	1.04950	0.013083	76.44	2.637
1.6	1.07751	0.01991	50.24	1.07548	0.016377	61.06	2.778
1.8	1.10357	0.02539	38.62	1.10181	0.02105	47.50	2.945
2.0	1.12941	0.03467	28.84	1.12700	0.02785	35.90	3.117
2.2	1.15513	0.04815	20.77	1.15254	0.03805	26.28	3.345
2.4	1.18067	0.06997	14.29	1.17793	0.05385	18.57	3.707
2.6	1.20597	0.10646	9.39	1.20816	0.07973	12.54	4.10
2.8	1.23111	0.1710	5.845	1.22826	0.12474	8.015	4.47
2.9	1.25606	0.2931	3.411	1.25322	0.2072	4.825	4.90
2.2	1.28212	0.5510	1.815	1.27802	0.3702	2.697	5.58
2.5	1.31752	1.679	0.5955	1.31495	1.053	0.945	6.48
2.7	1.34171	4.074	0.2455	—	(2.434)	0.411	7.2
2.9	1.37936 (19.9)	—	0.0503	—	—	—	—

Comparison with Results Obtained by Other Observers.—The only available observations on the viscosity of sucrose solutions at temperatures between 18° and 25° are those of: (i) Burkhard at 20°, as quoted by Lees (*Phil. Mag.*, 1901, [vi], 1, 128); (ii) R. Hosking at about 20° (*loc. cit.*, 285), and (iii) G. Rudolf at 25° (*Zeitsch. physikal. Chem.*, 1903, 43, 281).

Burkhard's results are quoted by Lees to three significant figures only.

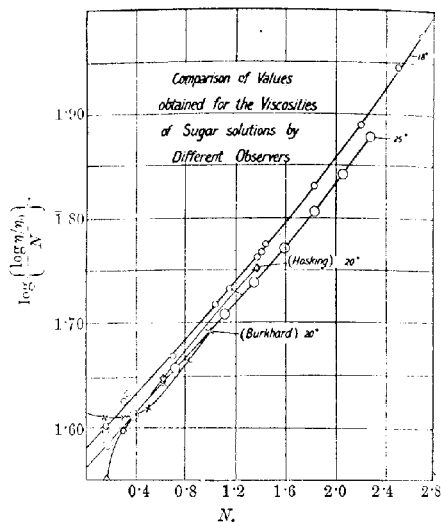
Hosking's experiments are very complete as regards the temperature range, measurements being made at intervals of 8° or 10° between 0° and 90°, but only five concentrations were examined. The viscosities used for comparison were observed within 0.25° of 20°, and are "interpolated values obtained by plotting the experimental results on a suitably large scale."

Rudolf's figures are obviously incorrect: in the case of the strongest solution, which is labelled 1.00*n*, the figure for the "Spec. Gew." corresponds almost exactly with the specific gravity of a seminormal solution of sucrose (1.06558), whilst " η " corresponds with the relative viscosity of a solution slightly weaker than 0.4*n*. It has been sug-

gested by Fawsitt (*Electrochemist and Metallurgist*, 1904, 3, 891), that the use of a spiral capillary gives abnormal values.

In order to show to what extent the formula $\frac{\eta}{\eta_0} = a^n$ (as proposed by Arrhenius, *Zeitsch. physikal. Chem.*, 1887, 1, 285) holds good for concentrated solutions, and because it is an accurate method of comparing different experimental values, the function $\log \left\{ \frac{\log \eta / \eta_0}{n} \right\}$ has been

FIG. 3.



calculated for each observation, and plotted against the corresponding value of n in Fig. 3.

The curves show that this function is not a constant, as it should be if Arrhenius's formula held good, and, further, they justify the conclusion that the results given in this paper may be taken as trustworthy.

The measurements also extend over a range of concentration nearly twice as great as that previously covered by other experimenters.

TABLE V.

Viscosity of Sucrose Solutions at 20°.—Comparison of Results
Obtained by Other Observers.

Per cent.	Barkhard.			Hosking.		
	η_{20}	η/η_0	$\log \left\{ \frac{\log \eta/\eta_0}{n} \right\}$	η	η/η_0	$\log \left\{ \frac{\log \eta/\eta_0}{n} \right\}$
0	—	—	—	0.01009	1.000	—
1	0.0293	—	—	0.01031	1.022	1.506
5	0.1488	1.15	1.601	0.01139	1.128	1.549
10	0.3035	1.33	1.6105	0.01328	1.316	1.5945
15	0.4645	1.56	1.6188	—	—	—
20	0.6323	1.89	1.6408	0.01910	1.893	1.6418
25	0.8068	2.35	1.6627	—	—	—
30	0.9887	3.07	1.6915	—	—	—
40	1.376	—	—	0.0607	6.013	1.7530

Viscosity and Conductivity of Aqueous Solutions of Hydrogen Chloride
and Lithium Chloride.

Materials Used.—(a) The hydrogen chloride solutions were obtained by suitable dilutions of the acid supplied by Merck as "guaranteed pure for analysis."

The concentration of each solution was determined by titration against a normal solution of sodium hydroxide, using methyl-orange as indicator. This solution had been checked against a standard solution of hydrogen chloride, the strength of which had been very accurately determined by the Iceland spar method (Masson, *Chem. News*, 1900, 81, 73; Green, *ibid.*, 1903, 87, 5).*

The density of each solution was not measured, as it was only required of moderate accuracy, but was taken from the published tables (at 15°) given by Ure after correction to 25° by Kremer's coefficients of expansion.

(b) The lithium chloride was obtained by dissolving an ordinary sample of lithium carbonate in hydrochloric acid; after filtration, the solution was evaporated to dryness. In this way, the excess of acid was driven off and a pure neutral salt obtained, which was dissolved in the minimum amount of water; the impurities, consisting mainly of alkaline sulphates, were salted out and separated by filtration.

A solution made up from this dry solid was 7.227*N* by weight of salt taken, and 7.218*N* by careful titration with a standard solution of silver nitrate.†

* This method was adopted as being the best available for the purpose; experience in this laboratory having shown that it is of a very high order of accuracy.

† Another proof of the efficiency of this method of purification is given by the

Occasionally the concentration of the solution under examination was determined by titration against a decinormal solution of silver nitrate, checked, in one instance, by evaporation to dryness; but the density of each solution was also observed at the temperature of experiment, and it was on this measurement that the determination of the concentration was mainly dependent. The following sets of published data on the density of lithium chloride solutions were available, and were therefore critically examined by the same method as was employed in the case of sucrose solutions.

1. Kremers (*Pogg. Ann.*, 1856, **99**, 435), at 19.5°.
2. Gerlach (*Zeitsch. anal. Chem.*, 1869, **8**, 279), at 15°.
3. Kohlrausch and Tollinger (*Wied. Ann.*, 1879, **6**, 38), at 18°.
4. Bender (*Wied. Ann.*, 1887, **31**, 872), at 15°.
5. Dijken (*Zeitsch. physikal. Chem.*, 1897, **24**, 81), at 15°.
6. Wade (Traus., 1899, **75**, 270), at 18°.
7. Conroy (*Proc. Roy. Soc.*, 1899, **64**, 312), at 18°.

The following new observations are recorded:

TABLE VI.

Density of Lithium Chloride Solutions.

At 18.0° (Orme Masson).			At 25.0° (W. H. G.).		
n .	d^{25}_4 .	$d_4 - d_w$	n .	d^{25}_4 .	$d_4 - d_w$
0.466	1.0098	0.02385	0.6159	1.01208	0.02356
0.937	1.0211	0.0239	0.9585	1.02000	0.02357
2.043	1.0464	0.02335	3.273	1.07173	0.02250
3.439	1.0768	0.02271	5.614	1.12229	0.02228
4.870	1.1078	0.02240	8.698	1.18640	0.02174
6.198	1.1352	0.02202	12.04	1.25680	0.02157
8.034	1.1748	0.02185	12.33	1.26364	0.02162
9.973	1.2154	0.02173			
12.227	1.2633	0.02164			

following analyses made by Professor Masson on the original stock solution of the purified salt:

Experiment.	Per cent. of chlorine in the solution.	Per cent. of lithium chloride in the solution.	Per cent. of chlorine in the lithium chloride.	Remarks.
I.	34.312	—	—	Gravimetric
II.	34.315	—	—	Volumetric
III.	34.305	41.110	83.446	Gravimetric

In experiment III, 2.576 grams were evaporated to dryness and yielded 1.669 grams of solid lithium chloride, in which, after re-solution in water, the chlorine was determined by precipitation with silver nitrate, 3.5742 grams of silver chloride being obtained. As the theoretical percentage of chlorine in lithium chloride is 83.451, the material used was of a high order of purity. The original solution was used several times over, the uncontaminated washings and diluted solutions being evaporated down again, as required, in a platinum basin. Traces of impurity were, however, salted out on each occasion, and were probably derived from the glass vessels in which the solution was stored and used.

Wade measured the contractions caused by diluting aqueous solutions with equal volumes of water, and then calculated the apparent molecular volumes of the salt itself. In the case of lithium chloride, his calculations are based on Kohlrausch's value of $d^{18.4} = 1.0274$ for a concentration $n = 1.209$, but he has erroneously taken this density as a "specific gravity" referred to water at the same temperature. I have recalculated his results, and they are now in excellent accord with the accurate measurements of Dijken for dilute solutions.

TABLE VII.
Dilute Lithium Chloride Solutions.

Wade (18°).			Dijken (16°).				
<i>n.</i>	$\frac{d_s - d_w}{n}$	ϕ .	<i>n.</i>	$d^{16.16}$	<i>n.</i>	$\frac{d_s - d_w}{n}$	ϕ .*
0.1	0.02485	17.652	0	1.000000	0	—	—
0.2	0.02468	17.822	0.23923	0.001405	0.0564	0.02489	17.61
0.3	0.02454	17.964	0.62360	0.003647	0.1472	0.00073	17.79
0.4	0.02442	18.081	1.01093	0.005921	0.2387	0.00071	17.81
0.5	0.02432	18.184					
0.75	0.02409	18.467					
1.0	0.02390	18.596					
1.209	0.02376	18.738					

* These figures have been recalculated.

All the above-mentioned data were plotted on the same* paper, and it was found that, within the limits of experimental error, the curves representing the variations of $\frac{d_s - d_w}{n}$ with concentration for temperatures varying from 15° to 25° are indistinguishable.

The density of solid lithium chloride at 25° has been carefully determined by Baxter (*Amer. Chem. J.*, 1904, **31**, 558) to be $D_{25}^{20} = 0.68$.

The table on p. 2038 gives the densities and molecular volumes calculated from the smooth curve drawn through the most trustworthy of the experimental results, and these values have been used throughout this research.

Apparatus.—The viscosity and density of each solution were measured exactly as described for sucrose solutions.

Conductivity.—The telephone method of Kohlrausch was employed for the determination of the electrolytic conductivity.

The cell used had, when filled with a "maximal" solution of magnesium sulphate, a resistance of about 200 ohms; one series of dilute solutions was, however, also examined in another cell, the resistance of which was only about 0.5 ohm for the same solution.

The conductivity of the distilled water used was 2.7 gemmhos.

Results.

Solutions of Hydrogen Chloride.—In 1901, a year before this research was started, Professor Masson had measured the conductivity of two

TABLE VIII.

Smoothed Values for the Density of Lithium Chloride Solutions.

n .	$\frac{d_s - d_w}{n}$.	$d^{18.4}_s$.	$d^{20.4}_s$.	Φ_{25} .
0.0	0.0250	0.99865	0.99712	17.53
0.1	0.02485	1.00114	0.99960	17.65
0.2	0.02470	1.00359	1.00206	17.83
0.5	0.02434	1.01082	1.00929	18.19
1.0	0.02390	1.02255	1.02102	18.63
1.5	0.02357	1.03400	1.03247	18.96
2.0	0.02330	1.04525	1.04372	19.24
3.0	0.02290	1.06735	1.06582	19.64
4.0	0.02255	1.08885	1.08732	19.99
5.0	0.02230	1.11015	1.10862	20.24
6.0	0.02210	1.13125	1.12972	20.44
7.0	0.02196	1.15237	1.15084	20.58
8.0	0.02186	1.17353	1.17200	20.68
9.0	0.02178	1.19467	1.19314	20.76
10.0	0.02171	1.21575	1.2142	20.83
11.0	0.02166	1.2369	1.2354	20.88
12.0	0.02162	1.2581	1.2566	20.92
12.5	0.02160	1.2686	1.2671	20.94
48.68*	0.02200	—	2.068	20.54

* Solid lithium chloride (Baxter, *loc. cit.*).

series of solutions of hydrogen chloride, and he has kindly handed over to me this previously unpublished work for inclusion in this paper. The results have been recalculated, and are included in tables IX and X.

TABLE IX.

Conductivity of Hydrogen Chloride Solutions at 24.9° (nominally 25.0°)
(by O. M., 1901).

Dilute Solutions.

n .	R .			$\Lambda \times 10$.
	Series a.	Series b.	Mean value (corrected).	
0	—	—	—	—
0.0001	—	220,000	220,000	0.4305
0.00025	—	87,000	87,000	0.4354
0.0005	—	44,800	44,800	0.4228
0.001	22,550	22,500	22,520	0.4205
0.0025	9,130	9,200	9,165	0.4133
0.005	—	4,700	4,650	0.4068
0.0075	3,100	—	3,100	0.4073
0.01	—	2,312	2,316	0.4059
0.025	939.5	940	939.4	0.4032
0.05	477.3	478	477.3	0.3968
0.075	322.3	—	322.0	0.3921
0.1	243.0	243	242.7	0.3862
0.25	100.7	—	100.4	0.3773
0.5	52.90	—	52.58	0.3602
0.75	36.85	—	36.53	0.3456
1.00	28.80	—	28.48	0.3325

CONDUCTIVITY OF SOME AQUEOUS SOLUTIONS. PART I. 2039

In order to make the author's determinations of viscosity as exactly comparable with these measurements as possible, the work was carried out at the same temperature, which was found, on accurately testing the thermometer, to be 24.90° (not 25.00° , as originally supposed). For the sake of completeness, the conductivity of each solution was also re-determined, and the results are given in table XI.

TABLE X.

Conductivity of Hydrogen Chloride Solutions at 24.9° (by O. M., 1901)
Concentrated Solutions.

n .	R .	λ .	Λ .
1.907	28.30	0.8346	0.3324
2.013	16.75	0.5654	0.2809
3.020	13.20	0.7174	0.2375
4.027	11.79	0.8032	0.1995
5.03	11.25	0.8418	0.1674
5.52	11.18	0.8470	0.1534
6.09	11.19	0.8463	0.1390
7.10	11.47	0.8256	0.1163
8.12*	11.98	0.7905	0.09735
8.182	12.01	0.7885	0.09637
9.187	12.68	0.7468	0.08129
10.264	13.58	0.6974	0.06795

* The strength of this solution, namely, $n=8.12$, was not very certain.

TABLE XI.

Viscosity and Conductivity of Hydrogen Chloride Solutions at 24.9° .

c .	$d^{25.4}$.	t .	η .	R (observed).	λ (corrected).	Λ .	$\Lambda\eta \times 10^3$.
0.001	0.9714	—	0.008977	—	—	—	—
0.005	1.0146	93.4	0.009518	28.92	0.3275	0.3290	3.131
0.01	0.0312	97.1	0.010082	17.00	0.5677	0.28125	2.835
0.02	0.0176	101.1	0.010692	13.55	0.7158	0.2361	2.467
0.05	0.0545	102.8	0.010952	12.74	0.7625	0.2199	2.4085
0.1	0.0716	107.5	0.011663	11.78	0.8300	0.1810	2.1106
0.2	0.0917	113.65	0.012595	11.54	0.8440	0.14295	1.8003
0.4	0.0923	113.7	0.012613	11.515	0.8458	0.14142	1.7836
0.7	0.1055	118.5	0.013324	11.725	0.8302	0.12190	1.6240
0.9	0.1153	124.35	0.014171	12.13	0.8012	0.1032	1.4623
0.95	0.1356	132.35	0.015347	12.825	0.7571	0.08489	1.3030
1.0	0.1570	144.5	0.017114	14.13	0.6857	0.06548	1.1207
1.5	0.1798	158.3	0.019160	15.48	0.6246	0.05218	0.9993

In tables XII and XIII are given the smoothed values obtained by plotting suitable functions of the above experimental values.

TABLE XII.

*Summary of Smoothed Results for Hydrogen Chloride.**Dilute Solutions: $\theta = 24.90^\circ$.*

<i>n.</i>	$\zeta/n.$	$\Lambda.$	<i>f.</i>	$\Lambda/f \times 10^3$
0.0001	0.0464	0.4525	111.40	4.055
0.00025	0.0630	0.4355	111.40	3.943
0.0005	0.0794	0.4255	111.40	3.829
0.001	0.1000	0.4200	111.40	3.779
0.0025	0.1357	0.4130	111.39	3.707
0.005	0.1719	0.4093	111.38	3.673
0.0075	0.1957	0.4074	111.36	3.656
0.01	0.2154	0.4065	111.33	3.652
0.025	0.2924	0.4023	111.23	3.617
0.05	0.3684	0.3972	111.07	3.579
0.075	0.4217	0.3936	110.92	3.542
0.1	0.4642	0.3905	110.74	3.525
0.25	0.6300	0.3783	109.75	3.447
0.5	0.7937	0.3617	108.14	3.344
0.75	0.9086	0.3470	106.56	3.256
1.000	1.000	0.3327	105.02	3.167

TABLE XIII.

*Summary of Smoothed Results for Hydrogen Chloride.**Concentrated Solutions: $\theta = 24.90^\circ$.*

<i>n.</i>	$\Lambda.$			<i>f.</i>	$\Lambda/f \times 10^3$
	O. M.	W. H. G.	Mean.		
1	0.3327	0.3289	0.3308	105.02	3.150
2	0.2841	0.2841	0.2841	99.16	2.865
3	0.2383	0.2380	0.2382	93.71	2.542
4	0.2005	0.2004	0.2005	88.61	2.263
5	0.1683	0.1681	0.1682	82.73	2.069
6	0.14125	0.14085	0.14105	78.96	1.7865
7	0.1184	0.11795	0.1182	74.18	1.5955
8	0.09942	0.09920	0.09931	69.45	1.439
9	0.08387	0.08373	0.08380	64.86	1.292
10	0.07101	0.07078	0.07090	60.44	1.173
11	—	0.06009	0.06009	56.18	1.0685
12	—	0.05192	0.05192	52.09	0.9965

Lithium Chloride.

A series of experiments on the conductivity of aqueous solutions of lithium chloride at 17.82° was also undertaken by Professor Mason, and the results given in table XIV are calculated from his observations. For comparison with these, the viscosity was measured by the author at the same temperature, and subsequently both the conductivity and viscosity were also measured at 25.00° in order to complete the work described in Part II of this research.

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It will be observed that here, as in the case of solutions of hydrogen chloride and sucrose, the strongest solutions obtainable have been worked with, the most concentrated being practically saturated.

TABLE XIV.

Conductivity of Lithium Chloride Solutions (by O. M.).

$\theta = 17.82^\circ$.

μ .	R (corrected).	$\kappa \times 10^4$.	Λ .
0.0028	1234.7	78.70	0.08260
0.166	285.2	332.0	0.07124
0.337	157.8	600.1	0.06404
1.539	108.1	876.0	0.05725
2.045	87.93	1077.0	0.05266
2.730	73.6	1287.0	0.04714
3.410	65.2	1452.5	0.04222
4.040	61.2	1547.0	0.03829
4.875	58.3	1624.3	0.03332
5.605	57.7	1641.2	0.02928
6.195	58.3	1624.3	0.02622
7.040	60.8	1555.0	0.02209
8.035	65.93	1436.4	0.01733
8.875	73.7	1285.0	0.01448
9.975	84.4	1122.0	0.011248
11.365	99.5	951.8	0.008614
12.25	120.63	785.0	0.006418
12.42	123.0	769.9	0.006199

TABLE XV.

Viscosity of Lithium Chloride Solutions.

$\theta = 17.92^\circ$.

μ .	$d17.82^\circ$.	t .	η .	$\log. \left\{ \frac{\log. \eta / \eta_0}{\mu} \right\}$.
0	0.99870	105.4	0.010630	—
0.411	1.03209	122.5	0.012872	2.76926
2.733	1.06267	143.2	0.01557	2.77495
3.415	0.08718	164.3	0.01834	2.78179
4.822	0.10858	187.3	0.021365	2.78959
5.654	0.12393	206.9	0.023964	2.79545
6.183	0.13510	225.8	0.02644	2.80617
6.320	0.13800	230.0	0.027005	2.80668
7.151	0.15565	263.8	0.031695	2.81912
7.941	0.17235	305.4	0.037027	2.83396
8.815	0.19178	368.4	0.04545	2.85481
9.973	0.21520	479.0	0.06030	2.87843
11.084	0.23867	640.0	0.08218	2.90333
12.096	0.25868	831.5	0.10851	2.92338
12.640	0.27136	991.8	0.13074	2.93568

TABLE XVI.
Conductivity of Lithium Chloride Solutions.
 $\theta = 25.00^\circ$.

n .	R (corrected).	$\lambda \times 10^4$.	Λ .	$\Lambda/f \times 10^4$.
0.00625	13800	6.86	0.1093	0.9797
0.0125	7100	13.34	0.1065	0.9553
0.025	3625	21.12	0.10436	0.9376
0.0499	1899	49.87	0.09990	0.9006
0.1000	982.7	96.37	0.09634	0.8746
0.2000	519.7	182.22	0.09110	0.8381
0.4000	279.4	338.9	0.08572	0.8009
0.6175	192.4	492.2	0.07971	0.7763
1.1630	114.7	825.6	0.07099	0.7430
2.001	76.8	1233.1	0.06162	0.7286
2.860	61.6	1537.3	0.05375	0.7155
4.029	52.7	1797.0	0.04460	0.7028
4.970	50.1	1890.2	0.03803	0.6916
5.325	49.73	1904.3	0.03576	0.6809
6.218	50.23	1886.3	0.03032	0.6806
6.976	51.9	1824.7	0.02616	0.6759
8.445	58.93	1607.0	0.01903	0.6628
8.804	61.33	1544.1	0.017539	0.6627
9.173	64.13	1476.7	0.016098	0.6636
10.400	75.9	1247.6	0.011997	0.6749
11.265	86.1	1100.0	0.009765	0.6876
12.345	101.1	936.7	0.007588	0.7276

TABLE XVII.
Viscosity of Lithium Chloride Solutions.
 $\theta = 25.00^\circ$.

n .	$d^{25}_w A$.	t .	η .	$\log \left(\frac{\log \eta \eta^0}{a - c} \right)$
0	0.99712	—	0.008955	—
0.6175	1.01213	95.6	0.009724	2.70288
1.163	1.02475	101.2	0.010466	2.76503
2.001	1.04376	111.7	0.011825	2.78057
2.860	1.06272	123.0	0.013312	2.77958
4.029	1.08794	141.6	0.015758	2.78606
4.970	1.10796	160.0	0.018188	2.79278
5.325	1.11554	168.6	0.019319	2.79733
6.218	1.03430	192.2	0.022444	2.80733
6.976	1.15032	217.5	0.025833	2.81333
8.445	1.18141	235.2	0.034829	2.84416
8.804	1.18897	307.3	0.037786	2.85139
9.173	1.19672	333.0	0.041229	2.85909
10.400	1.22264	444.2	0.05625	2.88501
11.265	1.24090	555.6	0.07144	2.90342
12.345	1.26364	732.0	0.09589	2.92122

As before, suitable functions were plotted, and the most probable values read off by interpolation from the smoothed curves.

The average temperature-coefficients of both the viscosity and conductivity for each concentration between 18° and 25° are tabu-

lated, and it will be seen that, although not identical, the ratio between them is practically constant for all concentrations.

TABLE XVIII.

Smoothed Values for the Conductivity and Fluidity of Lithium Chloride Solutions.

c	f		$\Lambda \times 10^2$		Percentage temperature-coefficients.		$\Lambda/f \times 10^4$	
	17°82°	25°00°	17°82°	25°00°	Fluidity.	Conductivity.	17°82°	25°00°
0	94·07	111·67	[9·888]	[11·53]	2·38	[2·38]	[1·047]	[1·0325]
0·0025	—	—	—	10·98	—	—	—	0·8796
0·0125	—	—	—	10·65	—	—	—	0·8553
0·025	—	—	—	10·436	—	—	—	0·8377
0·05	—	—	—	9·990	—	—	—	0·9007
0·1	92·82	110·16	8·215	9·634	2·38	2·22	0·8850	0·8745
0·2	91·59	108·70	7·768	9·110	2·37	2·22	0·8481	0·8381
0·5	87·98	104·35	7·052	8·228	2·37	2·14	0·8016	0·7885
1·0	82·20	97·46	6·281	7·312	2·36	2·11	0·7640	0·7501
1·5	76·78	90·98	5·757	6·710	2·35	2·13	0·7498	0·7374
2·0	71·66	84·87	5·306	6·163	2·35	2·08	0·7404	0·7261
2·5	62·26	73·64	4·517	5·258	2·34	2·11	0·7254	0·7138
3·0	58·84	63·65	3·849	4·476	2·32	2·10	0·7148	0·7022
3·5	46·21	54·59	3·262	3·785	2·32	2·07	0·7059	0·6920
4·0	39·18	46·27	2·728	3·170	2·31	2·11	0·6949	0·6850
5·0	32·68	38·60	2·227	2·604	2·32	2·17	0·6815	0·6745
6·0	26·65	31·55	1·803	2·105	2·34	2·15	0·6765	0·6672
7·0	21·23	25·28	1·422	1·6725	2·42	2·27	0·6696	0·6618
8·0	16·48	19·75	1·1166	1·3225	2·52	2·35	0·6776	0·6693
10·0	12·46	15·07	0·8710	1·0395	2·64	2·45	0·6897	0·6898
12·5	8·312	11·42	0·6794	0·8149	2·84	2·53	0·7295	0·7185
12·5	8·164	10·007	0·6003	0·7218	[2·88]	2·56	0·7352	0·7213

Comparison with other Published Values.—The only determinations of the viscosities of concentrated solutions of lithium chloride are those of Sprung (*Pogg. Ann.*, 1876, 59, 17) and the recent work of Hosking (*Phil. Mag.*, 1904, [vi], 7, 469), which, as in the case of his work on sucrose, covers a very complete range of temperature; but the interval between the different strengths examined is too large to admit of accurate interpolation, and the greatest concentration is $n=10·7$, whilst solutions of concentration up to $n=12·5$ may be readily prepared and worked with.

Hosking's results are slightly higher than those described here, but, on the other hand, his values for sucrose solutions were lower than mine.

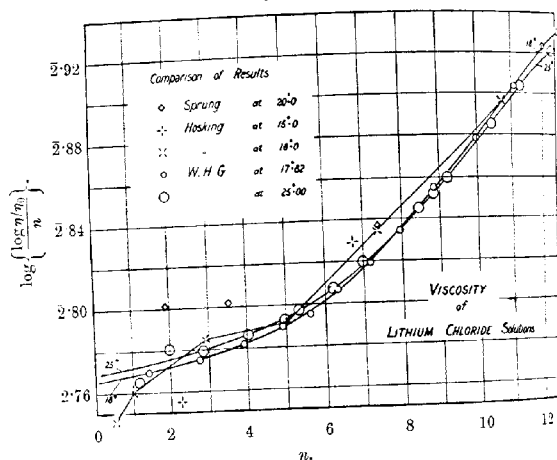
By plotting the function $\log. \left\{ \frac{\log. \eta / \eta_0}{n} \right\}$, it is possible to compare accurately the various sets of experiments (see Fig. 4).

TABLE XIX.

Viscosity of Lithium Chloride Solutions.—Comparison of Results by other Observers.

n .	p . per cent.	η .	η/η_0 .	$\log \left\{ \frac{\eta/\eta_0}{n} \right\}$.	Experimenter.
1.908	7.757	—	1.321	2.8008	Sprung, $\theta = 25^\circ$.
3.540	13.91	—	1.676	2.8018	
7.365	26.93	—	3.123	2.8370	
0.1030	—	0.01070	0.0041	2.600	Hosking, $\theta = 18.9^\circ$.
0.520	—	0.01133	0.0289	2.745	
1.012	—	0.01212	0.0582	2.760	
2.935	—	0.01600	0.1788	2.7848	
5.02	—	0.02169	0.3110	2.7921	
7.02	—	0.03365	0.5017	2.8341	
7.35	—	0.0739	0.8433	2.8966	
10.70	—	0.01530	0.1289	2.7550	
2.266	—	0.03256	0.4547	2.8290	
6.74	—	—	—	—	

FIG. 4.



Observations on the conductivities of lithium chloride solutions have been published by Kohlrausch, Fitzpatrick (dilute solutions), and other observers at 18° , and by Hosking at 18° and 20° , but the interpolation necessary to reduce their results so as to make them available for comparison with experiments at 17.82° and 25.00° does not readily admit of sufficient accuracy.

However, Hosking has also tabulated his values of $\frac{\Lambda}{f}$ at 18° and 20° and, as his readings were taken within about 0.2° of the latter temper-

ure, these figures furnish a trustworthy method of comparison, especially as $\frac{\Lambda}{f}$ varies only slightly with the temperature.

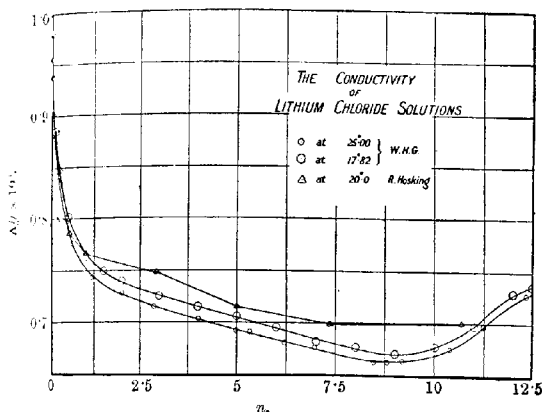
TABLE XX.

Conductivity of Lithium Chloride Solutions (by R. Hosking.)

$\theta = 20^\circ$.

κ	0.00645	0.1030	0.5198	1.0116	2.934	5.02	7.35	10.70
$\Lambda f \times 10^3$...	0.983	0.881	0.786	0.766	0.748	0.715	0.699	0.700

FIG. 5.



These results are plotted on Fig. 5 for comparison with the values of $\frac{\Lambda}{f}$ obtained by the author at 17.82° and 25.00° .

The discrepancy shown by the solution, which is nominally $2.934n_s$, both in this and in the viscosity curve, is probably due to some arithmetical error as to its concentration, especially as this was calculated merely from the density and not confirmed by analytical determinations.

Discussion of Viscosity Concentration Formulae.

The viscosity values given in this paper provide more complete means of testing various concentration formulæ than has hitherto been available, and they show that for dilute solutions, such as Arrhenius mixed with (not more than $0.2N$ -sucrose), that $\eta = A^x$ (where x = volume of solute per unit-volume of solution), or even $\eta = 1 + ax$ will represent the facts with fair accuracy; then for more concentrated

solutions, formulae of the type $\eta = A^x B^{1-x}$ are required, but even these soon cease to be accurate.

Reyher (*Zeitsch. physikal. Chem.*, 1888, **2**, 744), Wagner (*ibid.*, 1890, **5**, 31), Lauenstein (*ibid.*, 1892, **9**, 417), Kanitz, and others have come to similar conclusions.

Lees, in a valuable paper (*Phil. Mag.*, 1901, [vi], **1**, 128), shows by a comparison of the calculated with the observed values that neither the theoretically derived logarithmic, mobility, nor viscosity formula represents correctly the variation of viscosity with composition.

He then suggests the empirical relation :

$$\eta^n = v_1 \eta_1^n + v_2 \eta_2^n + \dots,$$

where v_1, v_2, \dots are the fractional volumes present of the several compounds, and η_1, η_2, \dots are their viscosities. This he found to hold good (within 1 per cent.) for a number of typical mixtures of liquids and solutions.

As n was found by Lees to have the value -0.5 for sucrose solutions, we may write the above formula as

$$\sqrt{f} = v_1 \sqrt{f_1} + v_2 \sqrt{f_2},$$

and we can simplify this to $\sqrt{f} = v_1 A + Bn$ if we take v_1 to denote the volume of water present per unit volume of solution, and v_2 to denote the volume of 100 per cent. sucrose solution (and not merely the volume of a 30 or 40 per cent. solution) per unit volume of the solution :

Now A is $\sqrt{f_0}$ (f_0 = fluidity of pure water),

and B is $\frac{\phi \sqrt{f_s}}{1000}$ (f_s = fluidity of a 100 per cent. solution of sucrose)

(ϕ = molecular volume of sucrose in solution).

Assuming Lees' formula to hold, B can be at once calculated from the viscosity at any concentration, for $B = \frac{\sqrt{f} - A v_1}{n}$.

B , thus calculated, varies from -2.35 at a concentration of $0.8n$ to -1.56 when $n=2.5$, as may be seen in column 4 of table XXI.

This indicates either that the fluidity of a 100 per cent. solution has a negative value, or that this formula also breaks down when rigidly tested.

An alteration in the value of the exponent of f will not materially improve the accuracy obtainable, nor can it lead to other than negative values for B , which is a simple function of the fluidity of a cent. per cent. solution of sucrose.

Lees' formula may, however, be thrown into another form ;
 thus, if $\sqrt{f} = v_1 \sqrt{f_0} + nB$,

then
$$\frac{\sqrt{f}}{f_0} = v_1 + n \frac{B}{\sqrt{f_0}}.$$

Now
$$v_1 = 1 - \frac{\phi}{1000}n.$$

$$\therefore \sqrt{\frac{f}{f_0}} = 1 - \left(\frac{\phi}{1000} - \frac{B}{\sqrt{f_0}} \right) n.$$

$$= 1 - \alpha n \text{ (where } \alpha \text{ is a constant),}$$

or
$$\frac{f}{f_0} = (1 - \alpha n)^2.$$

The degree of constancy of α can be seen from the values given in column 5 of table XXI.

TABLE XXI.

Fluidity of Sucrose Solutions at 25°.

Test of two forms of Lees' Fractional-Volumes Formula :

(i)
$$\frac{\sqrt{f}}{n} = \frac{\sqrt{f_0} v_1}{n} + B, \text{ where } \sqrt{f_0} = 10.567 = A.$$

(ii)
$$\frac{f}{f_0} = (1 - \alpha n)^2.$$

n .	$v_1 \times 10^3$.	\sqrt{f} .	$\sqrt{f/n} - \frac{Av_1}{n} = B.$	$\frac{1 - \sqrt{f/f_0}}{n} = \alpha.$
0	1000.0	10.567	—	—
0.1	978.9	10.120	-2.24	0.423
0.2	957.7	9.665	2.27	0.427
0.4	915.3	8.743	2.32	0.432
0.6	872.7	7.815	2.345	0.434
0.8	830.0	6.893	2.355	0.433
1.0	787.2	5.992	2.325	0.433
1.2	744.2	5.126	2.24	0.429
1.4	701.0	4.309	2.175	0.423
1.6	657.7	3.541	2.13	0.415
1.8	614.25	2.831	2.03	0.407
2.0	570.65	2.197	1.915	0.396
2.2	526.9	1.643	1.785	0.384
2.5	461.0	0.972	1.56	0.363

Many other formulæ have been tested, but the best results were obtained with an expression of the form $\frac{\eta}{\eta_0} = A^v w^w$, where v is the volume of the sucrose, and w is the volume of the water, present per unit-volume.

Varying discrepancies were observed according as the molecular volume of the water or the sucrose in the solution was assumed to be constant, and in no case was a very satisfactory concordance obtained between the calculated and observed viscosities.

This is in accord with Pickering (*Watt's Dictionary*, Vol. IV, *Solutions*, p. 492): "It may safely be stated that every attempt which has been made to express the properties of any series of solutions by a simple expression agreeing, within the limits of experimental error, with the observed values throughout a wider range of concentration has been unsuccessful, and that because the solvent cannot be regarded as inert and only playing the part of so much empty space."

Results and Conclusions.

1. The methods of standardising tubes for the measurement of viscosity have been discussed, and the values obtained for the viscosity of water by various observers have been critically examined and recalculated.

2. The viscosity-concentration curves for aqueous solutions of sucrose have been traced at 18° and 25°.

3. The values obtained for the density of lithium chloride solutions by previous observers have been compared and recalculated, and new observations added.

4. Measurements have been made of the conductivity and viscosity of aqueous solutions of hydrogen chloride at 25°, and of lithium chloride at 18° and 25°.

5. The figures obtained show that the connexion between viscosity and concentration is not accurately expressed, even within the limits of experimental error, by any of the various formulæ that have been suggested, but that the best concordance is obtained by the use of an

expression of the form $\frac{\eta}{\eta_0} = A^{v/w}$, where v = volume of solute, and w = volume of solvent, present per unit-volume of solution.

I desire to express my best thanks to Professor Orme Masson, at whose suggestion these investigations were undertaken, and who has kindly handed over to me previously unpublished measurements of the conductivity of hydrogen chloride and lithium chloride solutions for inclusion in this paper.

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VIII.—*Studies on the Viscosity and Conductivity of Some Aqueous Solutions. Part II. Mixtures of Solutions of Sucrose and Lithium Chloride. A Contribution towards the Elucidation of the Connexion between Ionic Mobility and the Fluidity of the Solution.*

By W. HEBER GREEN, D.Sc.

FROM the work of Faraday, Hittorf, Kohlrausch, Arrhenius, and van't Hoff, and, more particularly, from the direct measurements of ionic velocities in a jelly tube, we are driven to the conclusion that electrophoresis is a kind of convection of the ions into which the solute molecules are more or less completely dissociated, and also that each ion carries with it a definite surplus or deficiency of electricity which is given up to the electrode under the influence of an electromotive force above a certain limit.

Admitting therefore that these ions must be considered as moving through the solution under the influence of an electrical force, it follows that their velocity will be (a) strictly proportional to the potential slope between the electrodes, electrolytes obeying Ohm's law, and (b) dependent on the frictional resistance offered by the medium, which for such small bodies must be relatively very large, and their normal velocity will be reached almost immediately, just as fine sand grains fall through water at a uniform speed.

Such conclusions are independent of any theories as to the mechanism or completeness of ionisation, or as to any possible combination of the water with the solute molecules or ions; but the question arises whether this ionic resistance is identical with the ordinary physical or molar resistance as measured by Poiseuille's capillary tube method.

The relation between conductivity and fluidity has been studied by many experimenters, and their results may be summarised in the statement that, although there is, without doubt, a very close connexion between conductivity and fluidity, the difficulty of excluding other influences has made it impossible to determine the exact nature of that connexion.

The fact that a solidified jelly containing a salt in solution conducts almost as well as an ordinary aqueous solution of the same concentration, their conductivities are as 85:100, according to Arrhenius and Hagen, and, further, that there is no sudden change in the conductivity at the moment when the jelly sets, as shown by S. Arrhenius

(*Brit. Assoc. Rep.*, 1886, 344) and C. Ludeking (*Wied. Ann.*, 1888, 37, 172), indicates that in some cases at least the two fluidities (ionic and physical) are not identical; indeed, in such cases they may be compared, on the one hand, to the passage of water through the interstices of a fine sponge, and, on the other, to the sponge itself being forced to flow through a tube in such a way that no circumferential slipping takes place.

However, we find that many authors are inclined to assume a direct proportionality, and so quote the values of $\frac{\Lambda}{f}$ or $\frac{\Lambda_n}{\Lambda_o} \times \frac{f_o}{f_n}$ as "ionisation coefficients."

Thus, Bousfield and Lowry (*Phil. Trans.*, 1905, A, 204, 253), after pointing out that the viscosity of a 50 per cent. solution of sodium hydroxide is approximately seventy times as great as that of water, say: "This increase of viscosity must produce a large effect on the ionic mobility; the influence of this factor may be to some extent eliminated by dividing the molecular conductivity by the fluidity, and this ratio we have called the *intrinsic conductivity* of the solution."

This paper therefore describes an attempt to elucidate this question concerning the mechanism of conductivity, and it will be seen that, unless we are prepared to modify considerably our present day conceptions as to the ionisation of electrolytes, the evidence is strongly in favour of the velocities of the ions being approximately proportional to the two-thirds power of the fluidity, the exact relation probably depending on the relative sizes of the ions themselves and of the average interstitial spaces between the molecules of the solution.

The problem was attacked experimentally by adding a non-electrolyte to a solution of a good electrolyte so as to cause artificially a considerable decrease in the fluidity without materially affecting the ionisation of the salt. Simultaneous observations of both the conductivity and fluidity were made.

M. Wildermann (*Zeitsch. physikal. Chem.*, 1903, 46, 43) examined the influence of non-electrolytes on the degree of ionisation by means of the freezing-point method, and concluded that the addition of glycerol does not affect the ionisation of various organic acids.

The method under discussion has been previously used to a limited extent, notably by S. Arrhenius, Fawsitt, and Martin and Masson.*

Arrhenius did not find that the conductivity was proportional to the fluidity, but his results for the more dilute solutions (less than 10 per cent.) may be expressed by the formula $\Lambda = \Lambda_o \left(1 - \frac{a}{\eta}\right)^2$.

* The paper by Hartley, Thomas, and Applebey (*Trans.*, 1908, 93, 568) is discussed on p. 2060.

here y is the volume per cent. of non-electrolyte added, and α is a constant depending on the nature of both the salt and the non-electrolyte.

Fawcett (*Proc. Roy. Soc. Edin.*, 1903, 25, 51) studied the effect of additions of carbamide on the conductivity and viscosity of semi-normal solutions of hydrogen chloride, potassium chloride, and sodium hydroxide.

In the case of potassium chloride and sodium hydroxide, it will be seen that $\frac{\Lambda}{f}$ is very nearly a constant, indicating that the effect, under these conditions, of adding small amounts of carbamide is roughly proportional to the fluidity of the resulting solution.

The anomalous figures obtained in the case of addition of hydrochloric acid are probably due to the formation of carbamide hydrochloride.

Preliminary Investigation.

What may be regarded, for the purposes of this paper, as a series of preliminary experiments is to be found in a paper by C. J. Martin and Orme Masson (*Trans.*, 1901, 79, 707), who, while investigating the existence of certain complex ions, examined the influence of sucrose on the conductivities of solutions of potassium chloride, hydrogen chloride, and potassium hydroxide. They obtained evidence of salt formation in the last case, but with solutions of potassium chloride and hydrogen chloride no such complication occurred, and the diminution of conductivity may be fairly regarded as the effect of increased viscosity, with less possibility of disturbing influences than in any previous work on this subject.

As no determinations of the viscosity of these solutions were then available, no attempt was made to draw any inferences, such as might otherwise have been done. We are now, however, in a position to compare their results with the fluidity of pure sucrose solutions at 8°, which we may take as sufficiently correct for the purpose, as the viscosity effect of the small quantities of hydrochloric acid and potassium chloride present will be very small.

Reference to the figures, shows that Λ does not vary directly as f , but to some function more nearly approximating to $f^{\frac{2}{3}}$. The power 0.60 has been found to give the best results for the experiments with hydrochloric acid, and 0.70 when potassium chloride was the salt experimented on, that is :

$$\Lambda_f/\Lambda_{f_0} = \left(\frac{f}{f_0}\right)^{\frac{2}{3}}.$$

The actual velocities of the ions could only be directly proportional to the fluidity on the assumption that, apart from the viscosity effect,

the addition of sucrose to the solution so alters the dielectric constant, molecular association, or other physical property of the water as enormously to increase (to the extent of 400 per cent. in the most concentrated solutions) these ionic velocities.

What few facts have been observed would tend to lead us to expect a directly contrary effect, and these experiments, and those about to be described, clearly disprove the popular idea that the conductivity (corrected for imperfect ionisation) is proportional to the physical fluidity of the solution.

Scheme of Work.

The method involved in the above experiments of Martin and Masson has been extended, more attention being paid to experimental details, so as to cover as far as possible all ranges of concentration with an accuracy of about 0.1 per cent.

The salt chosen for the purpose was lithium chloride; its extremely great solubility (enabling concentrations of up to 12.5*N* to be readily obtained and worked with) making it especially suitable for the purpose.

Although the halides of sodium and ammonium were found by C. H. Gill (*Quart. Journ. Chem. Soc.*, 1857, 9, 269) to form crystalline compounds with sucrose, lithium chloride did not enter into such a combination, which might possibly have caused complications similar to those observed in the experiments of Martin and Masson with sodium hydroxide.

Concentrated solutions of lithium chloride are, however, open to the objection that they form complex ions, as shown by the observations of Hittorf and others on the migration constants, but this will not interfere with the deductions to be drawn from the behaviour of dilute solutions.

As it was thought that possibly inversion of the sucrose might be brought about by the presence of the lithium chloride, with a consequent small alteration of the viscosity, a solution was kept for some weeks and examined by a polariscope, but little or no inversion could be detected, and the effect of such a source of error may be dismissed as negligible.

This agrees with the observation of Bruner (*Zeitsch. physikal. Chem.*, 1900, 32, 133), that the hydrolysis of sucrose by lithium chloride is too small for measurement.

Besides the experiments on pure solutions of lithium chloride and sucrose, described in Part I, mixtures of these substances in three different ratios were examined, the viscosity, conductivity, concentration, and density being simultaneously observed for each solution. All observations were made at, or reduced to, 25.00°.

In series *A*, the ratio $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 0.05$.

A mixture was made up in which n (for LiCl) = 0.1 gram-mol. per litre of solution and N (for sucrose) = 2.0; this was diluted in successive stages until the conductivity could no longer be satisfactorily measured.

In series *B*, the ratio $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 0.5$.

The original mixture was $n = 1.0$ and $N = 2.0$, and was diluted similarly to the solutions in series *A*.

In series *C*, the ratio $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = 5.0$.

In this series, the strongest solution obtainable was approximately $n = 7.0$ and $N = 1.4$, for the presence of sucrose necessarily displaced considerable part of the water required for the solution of the lithium chloride. The viscosity of this mixture was, however, considerably greater than that of any solution in either of the other series.

Besides these three series, we have, at the one end, solutions of pure sucrose for which the ratio $\frac{n}{N} = 0$, and, at the other end, solutions of pure lithium chloride for which the ratio $\frac{n}{N} = \infty$. The details of these latter experiments are set out in Part I.

The concentration of each solution examined was determined by one or more of the following methods:

- (i) by titration. It was found that the presence of sucrose does not interfere with the titration of lithium chloride by a decinormal solution of silver nitrate, using potassium chromate as indicator.
- (ii) from the preparation of the solution: either from the weight of original substances taken or from the strength of previous solution, each dilution being quantitatively controlled.
- (iii) conversely to (ii): from strength of solution obtained by other quantitative dilution.
- (iv) from determination of density. For each series a smooth

curve was drawn through the points obtained by plotting $\frac{d_s - d_w}{n}$ against n , and this constituted a valuable check on the work, especially in the stronger solutions.

As an example, the concentrations given by the various methods available for series *C* are shown in table I.

The same materials and apparatus were used for measuring the density, fluidity, and conductivity of the mixtures as had been previously used in the case of the pure substances.

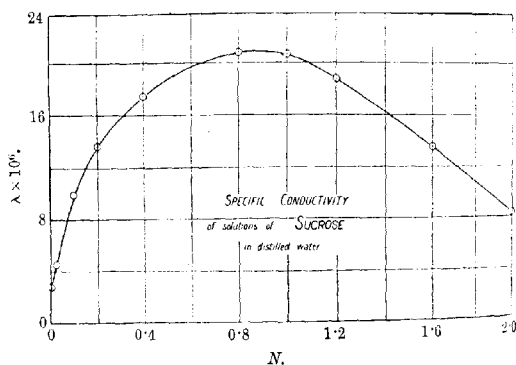
TABLE I.

Calculation of Normality of Lithium Chloride in each Solution.

$$\text{Series C: } \frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 5.$$

	By titration with N/20-AgNO ₃ .	By preparation from stronger solution.	From normality of diluter solutions.	From density.	Adopted value of n .
I.	7.218	—	—	7.194	7.218
II.	—	—	6.850	6.858	6.859
III.	—	5.802	5.832	5.833	5.832
IV.	5.054	5.028	5.054	5.054	5.054
V.	—	4.175	4.175	4.172	4.175
VI.	3.349	3.349	—	3.346	3.348
VII.	3.261	—	3.261	3.263	3.261
VIII.	2.471	2.471	—	2.473	2.471
IX.	—	—	1.834	1.833	1.833
X.	1.2578	—	1.2630	1.2588	1.2583
XI.	0.6855	0.6826	0.6872	0.6856	0.6856
XII.	0.3983	0.3982	0.3972	0.4005	0.3983
XIII.	—	0.2466	0.2459	—	0.2463
XIV.	0.1273	0.1277	0.1281	—	0.1278
XV.	—	0.06058	0.06093	—	0.06055
XVI.	0.03732	0.0370	—	—	0.03729
XVII.	0.01123	0.01113	—	—	0.0112

FIG. 1.



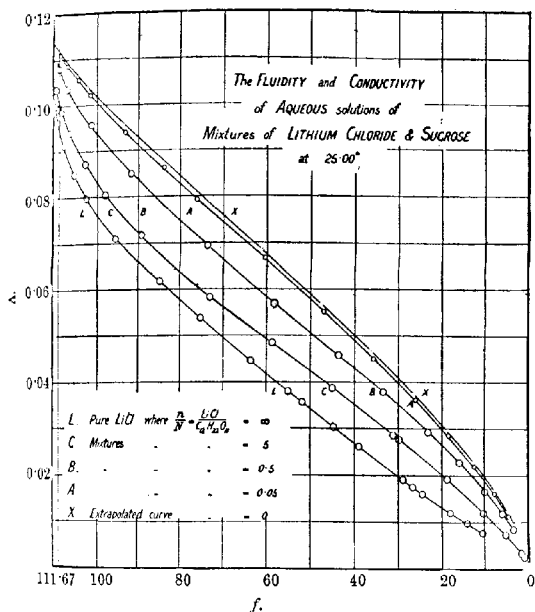
Series A was the first set of mixtures of sucrose and lithium chloride to be examined, and a fresh series of solutions was subsequently prepared and the measurements repeated, a low resistance cell (of the Arrhenius type with two movable electrodes) being used for the conductivity determinations.

CONDUCTIVITY OF SOME AQUEOUS SOLUTIONS. PART II. 2055

The observed conductivities were corrected by subtracting from them the conductivities found for the same concentrations of the sucrose alone, but, except for the weakest solutions, the corrections so made were insignificant.

Table II gives the observed conductivities of a series of sucrose solutions at 25.0°, and the general form of the curve (see Fig. 1), obtained by plotting the specific conductivity against the concentration,

FIG. 2.



similar to those cases in which conducting salts have been added to the sucrose solutions.

The residual conductivity shown by these solutions is probably due, at only to the small quantities of impurities present, but also to the small, but finite, ionisation of the sugar molecule itself, "for it is quite certain that sucrose is to be regarded as an extremely weak acid" (Martin and Masson, *loc. cit.*).

TABLE II.

The Conductivity of Solutions of "Brewers' Crystals" (Sucrose) in the Distilled Water Used.

$\theta = 25.00^\circ$.

<i>N</i> .	<i>R</i> ₂₅ v.	$\lambda \times 10^6$ (observed).	$\lambda \times 10^6$ (from smoothed curve).
2.0	56,000	8.43	8.4
1.8	—	—	10.9
1.6	34,500	13.7	13.7
1.4	—	—	16.3
1.2	25,000	18.9	18.9
1.0	22,700	20.8	20.8
0.8	22,500	21.0	21.0
0.6	20,600 (?)	22.9 (?)	19.9
0.5	—	—	19.0
0.4	27,000	17.5	17.7
0.3	—	—	16.1
0.2	34,300	13.77	13.8
0.1	47,000	10.05	10.0
0.04	—	—	6.2
0.02	105,000	4.54	4.5
Distilled water	175,000	2.70	2.7

EXPERIMENTAL.

The details of the experimental results obtained for pure lithium chloride, and for each of the three series of mixtures of sucrose and lithium chloride, solutions are given in the accompanying tables and plotted in Fig. 2.

TABLE III.

Fluidity and Conductivity of Solutions of Mixtures of Lithium Chloride and Sucrose.

$\theta = 25.00^\circ$.

Series A: $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 0.05$.

Con- ductivity cell used.	<i>d</i> ^{25.4} .	<i>n</i> .	<i>f</i> .	$\Delta \times 10$.	$\Delta f \times 10^3$.	$\Delta f \Delta c^{-1}$.
II.	0.99712	0	111.67	—	—	1.000
IIb.	0.99982	0.001005	109.65	1.1112	1.0134	0.195
IIb.	1.00246	0.002012	107.65	1.0862	1.0090	0.295
IIb.	1.01044	0.005020	101.91	1.0250	1.0058	0.295
IIc.	1.01055	0.005105	101.77	1.0205	1.0028	0.285
I.	1.02352	0.00996	93.26	0.9420	1.0100	0.285
IIb.	1.02368	0.01002	93.16	0.9380	1.0070	0.285
IIc.	1.0238	0.01006	93.01	0.9378	1.0083	0.285
IIa.	1.02416	0.01020	92.79	0.9359	1.0086	0.285
IIc.	1.03728	0.01517	83.96	0.8612	1.0259	0.285
I.	1.04981	0.01992	76.38	0.7947	1.0404	0.285
IIa.	1.05064	0.02024	75.80	0.7902	1.0424	0.285

TABLE III (continued).

Fluidity and Conductivity of Solutions of Mixtures of Lithium Chloride and Sucrose.

Con- ductivity Class.	$d^{25.4}$	n	f	$\Lambda \times 10$	$\Lambda/f \times 10^3$	$\Lambda/\Lambda_f = x$
I.	1.07631	0.03001	60.63	0.6704	1.1056	0.984
II.	1.07693	0.03025	60.10	0.6631	1.1036	
I.	1.10192	0.03982	47.27	0.5522	1.1682	0.978
II.	1.10324	0.04032	46.53	0.5501	1.1821	
II.	1.12436	0.04846	36.85	0.4659	1.2643	0.974
I.	1.12791	0.04982	35.67	0.4517	1.2663	
II.	1.12821	0.04994	35.575	0.4480	1.2593	0.975
II.	1.12850	0.05005	35.55	0.4508	1.2683	
I.	1.15402	0.05394	25.945	0.3612	1.3922	0.9775
II.	1.15408	0.05396	25.85	0.3619	1.4000	
II.	1.17716	0.06896	18.833	0.29135	1.5470	0.9775
I.	1.17909	0.06972	18.278	0.28485	1.5585	
I.	1.20428	0.07960	12.325	0.2182	1.7704	0.975
II.	1.20448	0.07968	12.313	0.2195	1.7827	
II.	1.22604	0.08822	8.463	0.1713	2.0245	0.979
I.	1.23040	0.08995	7.742	0.1608	2.077	
I.	1.23044	0.08995	7.722	—	—	0.975
II.	1.25253	0.09875	4.952	0.1208	2.4385	
I.	1.25475	0.09963	4.688	0.11503	2.454	0.975
II.	1.25346	0.09992	4.599	0.11288	2.455	

TABLE IV.

Fluidity and Conductivity of Solutions of Mixtures of Lithium Chloride.

$\theta = 25.00^\circ$.

Series B: $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 0.5$.

$d^{25.4}$	n (corrected).	f	$\Lambda \times 10$	$\Lambda \times 10^3/f$	$\Lambda/\Lambda_f = x$
1.02712	0	111.67	—	—	1.000
1.02993	0.01000	109.98	1.0815	0.9875	0.9655
1.02996	0.02016	107.74	1.0356	0.9640	0.944
1.01168	0.05000	101.42	0.9560	0.9446	0.926
1.02990	0.10000	91.52	0.8432	0.9211	0.8975
1.04456	0.1998	73.65	0.8459	0.9243	
1.02253	0.2989	57.75	0.6930	0.9424	0.880
1.11112	0.4003	43.53	0.5674	0.9840	0.8655
1.13607	0.4890	33.22	0.4618	1.0619	0.861
1.16614	0.5975	22.645	0.3795	1.1423	0.8605
1.18589	0.7000	15.305	0.2926	1.2905	0.8655
1.22086	0.7990	9.820	0.2228	1.4571	0.859
1.24799	0.8982	5.865	0.16565	1.6868	0.8575
1.25504	1.0004	3.219	0.11763	2.0076	0.860
			0.07901	2.4573	0.861

TABLE V.

Fluidity and Conductivity of Solutions of Mixtures of Lithium Chloride and Sucrose.
 $\theta = 25.00^\circ$.

Series C: $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = 5.0$.					
d^{25}_w	n (corrected).	f .	$\Lambda \times 10$.	$\Lambda \times 10^3/f$.	$\Lambda/\Lambda_f = x$.
0.99712	0	111.67	—	—	1.000
0.99778	0.01118	111.26	1.070	0.9618	0.941
0.99913	0.03720	110.30	1.016	0.9211	0.904
1.00036	0.06085	109.45	0.9860	0.9009	0.885
1.00368	0.1279	107.05	0.9398	0.8780	0.8635
1.00983	0.2463	102.94	0.8701	0.8453	0.831
1.01755	0.3983	97.90	0.8035	0.8207	0.8045
1.03188	0.6856	89.13	0.7170	0.8045	0.7795
1.06016	1.2583	72.97	0.5883	0.8062	0.7525
1.08798	1.833	58.60	0.4851	0.8278	0.732
1.11839	2.471	44.84	0.3876	0.8648	0.710
1.15520	3.261	30.96	0.2863	0.9247	0.6825
1.15914	3.348	29.535	0.2762	0.9352	0.680
1.19702	4.175	18.51	0.19105	1.0320	0.649
1.23692	5.054	10.062	0.1199	1.1917	0.6115
1.27200	5.832	5.121	0.07221	1.4101	0.578
1.31778	6.850	1.601	0.03174	1.982	—
1.33258	7.218	0.942	0.02329	2.473	—

TABLE VI.

Fluidity and Conductivity of Solutions of Pure Lithium Chloride.
 $\theta = 25.00^\circ$.

Series L: $\frac{\text{LiCl molecules}}{\text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}} = \frac{n}{N} = \infty$.					
n .	d^{25}_w .	f .	$\Lambda \times 10$.	$\Lambda/f \times 10^3$.	$\Lambda/\Lambda_f = x$.
0	0.99712	111.67	—	—	1.000
0.00625	0.99727	111.57	1.098	0.9796	0.961
0.0125	0.99743	111.48	1.065	0.9553	0.937
0.025	0.99774	111.28	1.0436	0.9377	0.920
0.05	0.99836	110.90	0.9990	0.9007	0.8845
0.1	0.99960	110.16	0.9684	0.8745	0.8575
0.2	1.00206	108.70	0.9110	0.8381	0.821
0.5	1.00929	104.35	0.8228	0.7885	0.7745
1.0	1.02102	97.46	0.7312	0.7501	0.735
1.5	1.03247	90.98	0.6710	0.7374	0.717
2.0	1.04372	84.87	0.6163	0.7261	0.6955
3.0	1.06582	73.64	0.5253	0.7138	0.6665
4.0	1.08732	63.65	0.4476	0.7032	0.635
5.0	1.10862	54.59	0.3785	0.6920	0.601
6.0	1.12972	46.27	0.3170	0.6850	0.568
7.0	1.15084	38.60	0.2604	0.6745	0.531
8.0	1.17200	31.55	0.2105	0.6673	0.495
9.0	1.19314	25.28	0.16725	0.6618	0.4595
10.0	1.2142	19.75	0.13225	0.6693	0.4305
11.0	1.2354	15.07	0.10395	0.6898	0.4055
12.0	1.2566	11.42	0.08149	0.7135	0.382
12.25	1.2671	10.007	0.07218	0.7213	0.3695

It might be objected that Λ or $\frac{\Lambda}{f}$ for each series should have been plotted against the concentration of either the lithium chloride or the sucrose, instead of against the fluidity, as has been done, but the various curves would not then have been so readily comparable, nor their whole range so nearly completed, as has been possible, but in any case it is obvious that in no set of solutions does $\frac{\Lambda}{x}$ vary directly as f , although the effect of x , the ionisation factor of the salt, being unknown must to some extent obscure the question at issue.

As, for any given fluidity, there are four solutions each containing a different ratio of lithium chloride to sucrose molecules present, it is possible to construct a curve by plotting $\frac{\Lambda}{f}$ against $\frac{n}{n+N}$, which, passing through these four points, can be accurately extrapolated to the axis where $\frac{n}{n+N}$ is zero. This operation can be repeated for as many different fluidity values as we please, and thus we may obtain a series of values of $\frac{\Lambda}{f}$ for solutions of various fluidities (and therefore sucrose concentrations), but of infinite dilution as regards the lithium chloride.

In order to eliminate errors much exceeding one part per thousand in these final results, it was necessary to plot various more or less complex functions; thus, in the extrapolation to infinite dilution, the complex quantity $\log \frac{\Lambda}{f^{0.7}} + \frac{n}{n+N}$ was plotted against $\frac{n}{n+N}$.

Thus the effects of incomplete ionisation are eliminated, and the dependence of conductivity on viscosity, with a minimum of other interfering influences, can be examined.

The numerical values are given in table VII, and plotted as curve X in Fig. 2.

It will be observed that Λ is by no means proportional to f , but more nearly to $f^{0.7}$, although even when $f=0$ the values of Λ do not appear to extrapolate to a zero value. This is especially noticeable in series C, for which $\frac{n}{N}=5$, and where some extremely viscous solutions were examined.

* Dividing Λ by $f^{0.7}$ has brought the various curves closer together, so that they could all be plotted on the same sheet, and using the logarithm ensures an equal percentage accuracy throughout; the second term merely alters the slope of the curves, making them more nearly horizontal and permitting an increased magnification of the scale used in plotting.

TABLE VII.

Extrapolation of the Conductivities of Mixtures of Sucrose and Lithium Chloride to the Ratio $\frac{\text{LiCl}}{\text{Sucrose}} = 0$.

The conductivities in each series corresponding with the fluidities of solutions containing "*N*" gram-molecules of sucrose only are as follows:

Sucrose, $n = 0$.		$\text{LiCl} + \text{C}_{12}\text{H}_{22}\text{O}_{11}$	$\text{LiCl} + \text{C}_{12}\text{H}_{22}\text{O}_{11}$	$\text{LiCl} + \text{C}_{12}\text{H}_{22}\text{O}_{11}$	$\text{LiCl} + \text{C}_{12}\text{H}_{22}\text{O}_{11}$	LiCl + $\infty \text{C}_{12}\text{H}_{22}\text{O}_{11}$, Extrapolated series in which $n/N = 0$.		
<i>N</i> .	<i>f</i> .	$n/N = \infty$, $\Lambda \times 10^3$	$n/N = 5$, $\Lambda \times 10^3$	$n/N = \frac{1}{2}$, $\Lambda \times 10^3$	$n/N = \frac{1}{3}$, $\Lambda \times 10^3$	$\Lambda_0 \times 10^3$	$\frac{\Lambda_0 f}{\Lambda \times 10^3}$	$\frac{\Lambda_0 f}{\Lambda \times 10^3}$
0	111.67	—	—	—	—	114.0	—	—
0.02	109.75	94.6	99.7	107.5	111.2	111.84	1.019	4.79
0.05	107.00	87.0	93.75	102.5	107.85	108.85	1.017	4.52
0.1	102.40	79.15	86.15	96.0	102.9	104.15	1.017	4.05
0.2	93.42	69.0	75.6	86.1	94.15	95.80	1.025	4.00
0.4	76.43	54.85	61.5	71.45	79.5	81.05	1.060	3.86
0.6	61.06	42.73	50.25	59.3	67.1	68.67	1.125	3.66
0.8	47.51	32.55	40.60	49.1	55.6	56.85	1.197	3.41
1.0	35.91	24.15	32.25	40.20	45.50	46.62	1.298	3.09
1.2	26.28	17.41	25.15	32.30	36.54	37.38	1.422	3.04
1.4	18.57	12.48	19.15	25.45	28.83	29.50	1.588	3.15
1.6	12.54	8.86	14.20	19.50	22.20	22.72	1.812	3.47
1.8	8.017	—	10.08	14.45	16.45	16.83	2.099	3.90
2.0	4.826	—	6.91	10.35	11.77	12.02	2.490	3.96
		$n/n + N$ $= 1.0$	$n/n + N$ $= 0.833$	$n/n + N$ $= 0.333$	$n/n + N$ $= 0.0475$	$n/n + N = 0$		

Hartley, Thomas, and Applebey (*loc. cit.*) have recently measured the effect of additions of pyridine on the conductivity and viscosity of dilute solutions of lithium nitrate, and their results are of especial interest in this connexion.

They have interpreted their results and calculated the ionisation coefficients of the lithium nitrate on the assumptions: (1) that Stokes' theorem can be applied to bodies of molecular size, and consequently that ionic mobility is proportional to physical fluidity; (2) that each ion is accompanied by an aqueous envelope; and (3) that the diameter of this envelope varies with the percentage of pyridine present, but not appreciably with the non-ionised lithium nitrate in the solution.

Before accepting the conclusions of these authors, it must be borne in mind that: (1) it is extremely doubtful whether Stokes' theorem holds good where the moving body is of the same order of dimensions as the particles of the medium, and, further, the experiments described in this paper indicate that the ions will still have some mobility even when the fluidity of the solution as a whole has become zero; (2) the assumption of the existence of an aqueous envelope around each ion

rests on very indefinite evidence, and is difficult to reconcile with the conductivity of solidified jellies and similar phenomena; and (3) the lithium nitrate, in so far as it is non-ionised, is as essentially a part of the solvent as the pyridine or the water, and therefore its presence must affect the nature and size of the envelopes, if such, indeed, do exist.

The peculiar physical properties of pyridine mixtures, and the small variation of viscosity observed, whilst rendering the results obtained of interest in other directions, indicate a serious drawback to its use as a means of testing the connexion between ionic mobility and physical fluidity. Sucrose, on the other hand, has been chosen as especially suitable, both on account of its enormous effect on the viscosity (a 2*N*-solution has only about 4 per cent. of the fluidity of pure water) and on account of the comparative simplicity of its other physical properties.

Disarding, for the time being, the theory of aqueous envelopes, my results afford another method for the calculation of ionisation-coefficients. If we compare the conductivity (Λ) of a pure lithium chloride solution with the conductivity (Λ_f) of a solution infinitely dilute with respect to its lithium chloride content, but which contains sufficient sucrose to give it the same fluidity as the first solution, then the coefficient of ionisation should be given by $\frac{\Lambda}{\Lambda_f} = \alpha$.

This calculation has been made for pure lithium chloride and also for the various mixtures of lithium chloride and sucrose examined, and the results have been tabulated in the last columns of tables III—VI and plotted in Fig. 3.

Comparison of the curves seems to show, assuming that the mobility effect has been eliminated, that the addition of sucrose enormously increases the ionisation of the lithium chloride. But such a conclusion is unnecessary, for if we regard each ion as an independent particle threading its way between the molecules of which the solution is composed, then mobility at any instant will not depend on the fluidity of the solution, but on the possibility of the ion finding a pore-space through which to pass the section of liquid immediately in front of it, and this, if the dimensions of the ion compared with the pore-spaces be negligible, will be proportional to the two-thirds power of the total free space per unit-volume of solution. If the ionic sizes be larger or the interstitial spaces smaller, then a higher and varying power will be required to express the relation between them.

Consequently, neither the method used by Hartley, Thomas, and Applebey nor that detailed above can be expected to give the true ionisation-coefficients, for the ionic mobilities in solutions of the same fluidity will vary according as the viscosity is mainly due to lithium salt, sucrose, or pyridine.

Whilst the exact connexion between the fluidity and the structure of a liquid is unknown, these ideas cannot be tested quantitatively, but they are in qualitative agreement with the experimental results collected in table VIII.

FIG. 3.

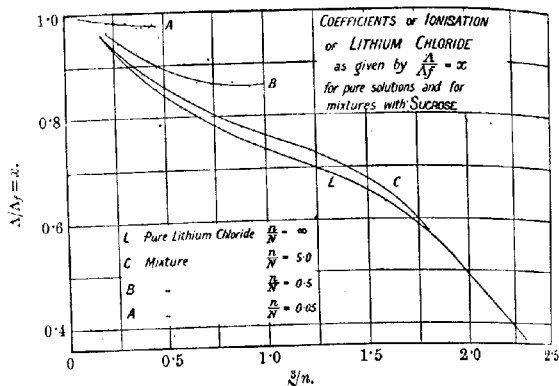


TABLE VIII.

Summary of Results Obtained with Different Electrolytes and "Viscosity-producers."

Experimenter.	Electrolyte.	Non-electrolyte used to increase viscosity.	Function of fluidity approximately proportional to mobility.
Martin and Masson..	{ HCl KCl	Sucrose	$f^{0.45}$
W. H. G.	LiCl	"	$f^{0.70}$
Massoulier *	CuSO ₄	Glycerol	$f^{1.0}$
Fawsitt	{ NaOH KCl	Carbamide (dilute solutions)	$f^{1.0}$
Hartley, Thomas, and Applebey	LiNO ₃	{ Pyridine (up to 65 per cent.)	$f^{1.0}$ to $f^{1.2}$

* *Compt. rend.*, 1900, 130, 773.

Summary.

1. Measurements have been made at 25° of the conductivity and viscosity of aqueous solutions of three series of mixtures of lithium chloride and sucrose, in which the ratio LiCl molecules : C₁₂H₂₂O₁₁ molecules was 1 : 0.2, 1 : 2.0, and 1 : 20 respectively.

2. By extrapolation from the results for the above mixtures, the conductivity and viscosity values were calculated for a series in which the ratio $\text{LiCl molecules} : \text{C}_{12}\text{H}_{22}\text{O}_{11} \text{ molecules}$ was $1 : \infty$. In this way, the variations in the ionic mobilities at infinite dilution of the salt, concomitant with change of fluidity due to the presence of sucrose in solution, were rendered free from some, if not all, disturbing factors.

3. The ionic mobility is not simply proportional to the physical fluidity of the solution, but more nearly to the two-thirds power in the case of lithium chloride and sucrose; with other ions and other viscosity-producing molecules, the exact function varies, and presumably depends on the relative molecular sizes.

4. No trustworthy method has as yet been established whereby the true ionisation-coefficients of a salt in solution can be determined.

My best thanks are due to Prof. Masson, not only for many valuable suggestions, but also for the great interest he has shown throughout these investigations.

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CV.—*The Formation of Polyiodides in Nitrobenzene Solution. Part IV. The Electrolytic Dissociation of the Polyiodides of the Alkali Metals and Ammonium Radicles.*

By HARRY MEDFORTH DAWSON and COLIN GYRTH JACKSON.

As a result of the investigation of the distribution of iodine and polyiodides between water and nitrobenzene, an account of which has been already published (Dawson, *Trans.*, 1908, **93**, 1308), the conclusion was drawn that soluble polyiodides of the alkali metals and ammonium salts, represented by the general formulæ MI_3 , MI_5 , MI_7 , and MI_9 , are formed in nitrobenzene solution as the proportion of iodine to alkali or ammonium salt in such solutions is gradually increased. From the phenomena of distribution a considerable amount of information relative to the chemical dissociation of the polyiodides was obtained, and this has now been supplemented by the investigation of certain physical properties, the results of which throw a good deal of light on the electrolytic dissociation of the polyiodides in nitrobenzene solution.

In particular, measurements have been made of the freezing

temperatures and of the electrical conducting powers of polyiodide solutions. Further, from density determinations, numbers have been obtained which represent the molecular solution volume of the dissolved polyiodides, and from transport measurements it has been possible to calculate the relative velocities of certain ions in nitrobenzene solution.

Preliminary mention should be made of the fact that, in certain cases, it has been found more convenient to make up solutions for comparison in such a way that they contain equimolar quantities of dissolved substance in unit volume of the solvent, instead of, as is more usual, in unit volume of the resulting solution. Such solutions are not "molar" solutions in the ordinary sense of the word, and when this method of preparing comparable solutions has been used, special reference will be made to the fact. When very dilute, the two series of solutions are, of course, practically identical.

Freezing-point Measurements.

In the first part of the paper, an account of the results of freezing-point determinations will be given.

The use of nitrobenzene as a solvent for cryoscopic measurements presents certain difficulties. Augustin (*Diss.*, Leipzig, 1908) found gradually decreasing values for the freezing-point constant when successive quantities of benzil were added to freshly-distilled nitrobenzene. This observation has been explained by Beckmann and Leckmann (*Zeitsch. physikal. Chem.*, 1907, **60**, 385) as being due to the gradual absorption of water by nitrobenzene (compare also Hansen, *ibid.*, 1904, **48**, 593), and these observers showed that the freezing-point constant, which is less than 70 for moist nitrobenzene, increases to more than 80 when perfectly dry nitrobenzene is used as a solvent. On account of this circumstance, considerable care must be exercised when nitrobenzene is used for molecular-weight determinations.

In the experiments to be described, we endeavoured to eliminate the influence of absorbed water by making comparative experiments on series of solutions, all of which were made up from the same sample of nitrobenzene which had been exposed to the air for several days. The solutions were prepared in exactly the same manner, and were kept in well-fitting stoppered bottles up to the moment at which they were introduced into the freezing-point apparatus. This consisted of a cylindrical tube, fitted with a perforated stirrer, which could be rotated rapidly about a vertical axis round the Beckmann thermometer, and, further, with a short side-tube, in which was inserted a glass rod carried by a cork closing the tube. By removing the glass rod, bringing it into contact with a small quantity of solid

nitrobenzene cooled to -20° , and quickly replacing it, crystallisation would be at once induced when the desired degree of supercooling had been obtained. Precautions were taken to ensure that the rate of rotation of the stirrer and the degree of supercooling were the same in all the experiments of a series. Solutions containing 0.1 molar quantities of iodine and potassium iodide, corresponding with the formulae KI_3 , KI_5 , KI_7 , and KI_9 , 100 c.c. of nitrobenzene were prepared, and the freezing points of these, as well as of the nitrobenzene used as solvent and an equivalent solution of iodine, were then determined. On the following day, the freezing points of the various liquids, which had in the meantime been stored in the stoppered bottles already referred to, were again determined. Table I gives the two series of freezing-point depressions, the means of the corresponding values, and the relative depression with reference to the iodine solution which may be regarded as the standard.

TABLE I.

Solution.	Freezing-point depressions.		Mean depression.	Relative depression.
	0.567°	0.575°		
I_2	1.036	1.027	1.031	1.00
KI_3	1.088	1.075	1.082	1.90
KI_5	1.274	1.291	1.282	2.25
KI_7	1.572	1.557	1.564	2.74

A second set of determinations made with specially prepared solutions gave the following series of values for the relative freezing-point depressions: 1, 1.85, 1.90, 2.27, and 2.71, whilst a third set of experiments gave the series: 1, 1.84, 1.90, 2.25, and 2.70. The mean values are recorded in table II.

TABLE II.

Relative Freezing-point Depressions of Polyiodide Solutions.

I_2	KI_3	KI_5	KI_7	KI_9
1	1.83	1.90	2.25	2.72

The figures in the table may be regarded as representing the numbers of molecules in the various polyiodide solutions relatively to the number in the equivalent iodine solution, for which the cryoscopic behaviour has been shown to be normal, the molecular weight corresponding with the formula I_2 . Now the concentration of the free iodine in 0.1 molar solutions of the polyiodides has been determined by distribution measurements (*loc. cit.*); the values for the tri-, penta-, hepta-, and enne-iodide solutions at 18° are

0.000016, 0.0030, 0.043, and 0.103 mol. per litre respectively. For the freezing-point measurements, solutions containing 0.1 molar quantities of the polyiodides per litre of nitrobenzene were used, and for these the free iodine concentrations are slightly smaller, namely, 0.000016, 0.0049, 0.042, and 0.100 mol. per litre respectively. It is assumed here that the chemical dissociation is practically the same at the freezing points of the solutions as at 18°. According to these figures the chemical dissociation of the tri-iodide is negligibly small, and the relative freezing-point depression of 1.83 must be attributed to electrolytic dissociation, the extent of which, according to the equation $\alpha = i - 1/n - 1$, is 83 per cent. The tri-iodide is therefore very considerably ionised, and this result is in agreement with the high electrical conductivity of the solution.

The chemical dissociation in the penta-, hepta-, and ennea-iodide solutions is far from negligibly small, and it remains to be seen whether the calculated concentrations of free iodine are such as will explain the greater freezing-point depressions observed in the case of these solutions. Assuming that the degree of electrolytic dissociation is the same in the several polyiodide solutions, the relative freezing-point depressions, calculated on the basis of the above values for the free iodine concentrations, are for the penta-, hepta-, and ennea-iodide solutions: 1.88, 2.25, and 2.83, whereas the observed values are 1.90, 2.25, and 2.72. By taking the chemical dissociation into consideration, it is therefore possible to account satisfactorily for the greater freezing-point depressions of the higher polyiodide solutions when the several polyiodides are assumed to be equally ionised.

In order to obtain confirmatory evidence of the general agreement between the results of freezing-point and distribution measurements, and to test the conclusion drawn in the previous paper, that the chemical dissociation of the polyiodides is independent of the contained alkali metal or ammonium radicle, a second series of freezing-point determinations was carried out with solutions of tetramethylammonium polyiodides. The relative freezing-point depressions in table III represent the mean values obtained in two series of experiments, and the calculated values for solutions of the penta-, hepta-, and ennea-iodides are based, as before, on the assumption that the degree of electrolytic dissociation of these is the same as that of the tri-iodide.

TABLE III.

0.1 mol. per litre nitrobenzene	I_3	NI_3	NI_5	NI_7	NI_9
Relative freezing-point depression ...	1	1.77	1.89	2.20	2.65
Calculated relative depression	—	—	1.82	2.19	2.77

The agreement between the observed and calculated values is not so good as in the case of the potassium polyiodide solutions, but it is sufficient to indicate that, in general, the increase in the freezing-point lowering which is observed on passing from the penta- to the ennea-iodide solution can be approximately accounted for by the increase in the extent of the chemical dissociation.

In both the above series of measurements, a comparison of the simple iodide with the polyiodides was impossible by reason of the insolubility of the iodide in nitrobenzene. This comparison appeared to be of considerable interest, and, since tetrapropylammonium iodide dissolves readily in nitrobenzene, it was possible to compare the freezing points of corresponding solutions of iodine, tetrapropylammonium iodide, and the tri-iodide. As in the previous series, the solutions were made by dissolving 0.1 molar quantities in a litre of nitrobenzene, and the freezing-point depressions, obtained from two independent series of experiments, were found to be in the ratio 1:1.35:1.68. It is evident that the freezing-point lowering for the tri-iodide solution is considerably greater than that for the equivalent solution of the iodide, and, since the chemical dissociation of the tri-iodide is negligibly small, this can only be referred to a difference in the extent to which the iodide and tri-iodide are electrolytically dissociated. The two lowerings correspond with 35 and 68 per cent. ionisation for the iodide and tri-iodide solutions respectively.

Corresponding with the much greater osmotic concentration, it was anticipated that the electrical conductivity of the tri-iodide solution would be greater than that of the solution of the iodide. On examination of the two solutions, the specific conductivities at 18° were found to be 12.73×10^{-4} and 16.29×10^{-4} for the iodide and tri-iodide respectively. The tri-iodide solution is therefore, as anticipated, a much better conductor than the corresponding solution of the iodide.

Electrical Conductivity Measurements.

The fact that nitrobenzene solutions of the polyiodides are good electrical conductors has been already indicated in a previous paper (Dawson and Gawler, *Trans.*, 1902, 81, 524). From the few measurements made at that time, it was not possible to draw any general conclusion beyond that the molecular conductivity increases with the dilution. The results of a series of comparative measurements of the conductivities of solutions of the polyiodides of the alkali metals and certain ammonium bases are, however, now available. Special experiments showed that the conductivity of a given solution depends to a small extent on the quantity of absorbed water. but

since comparative measurements only were required, no attempt was made to determine the conductivities in perfectly dry nitrobenzene, and the solvent used consisted of nitrobenzene which had been exposed to the atmosphere of the laboratory for several days. In table IV the specific conductivities of solutions are recorded, which correspond with the formulæ MI_3 , MI_5 , MI_7 , and MI_9 , and contain 0.1 mol. polyiodide per litre of nitrobenzene.

In general, these solutions were prepared by weighing out corresponding quantities of iodide and iodine into stoppered bottles, and adding from a burette the amount of nitrobenzene required to give solutions of the requisite concentration. On the other hand, the tri-iodide solutions of sodium, potassium, rubidium, cesium, ammonium, and tetramethylammonium were obtained by saturating a previously prepared iodine solution with the respective iodides, solubility determinations having shown that these saturated solutions correspond almost exactly with the tri-iodide formula.

TABLE IV.

Specific Conductivities of Polyiodide Solutions. $\kappa 10^4$.

Iodide.	MI_3 .	MI_5 .	MI_7 .	MI_9 .
NH_4I	22.50	22.47	21.68	21.20
RbI	23.30	22.25	21.22	20.71
KI	23.38	21.47	20.88	20.23
CsI	22.65	21.37	20.41	19.65
NaI	22.94	21.10	19.96	19.38
NMe_4I	21.03	19.77	19.04	18.51
LiI	20.74	18.89	18.05	17.58
NEt_4I	19.36	18.04	—	—
NPr_4I	16.29	15.14	14.60	14.31

It will be observed that the numbers in the last three columns decrease regularly from the top to the bottom of the table, and this relationship has determined the order in which the various iodides are arranged in the table.

The conductivities of the first five tri-iodide solutions, which, as already mentioned, were prepared by saturating a nitrobenzene solution containing 0.1 mol. iodine per litre of solvent with the respective iodides, do not follow the same order. It was naturally supposed that this difference in order might be due to differences in the solubility of the iodides in nitrobenzene, and information was sought by measurement of the conductivities of the solutions obtained by agitating the finely powdered iodides with nitrobenzene at 18° for about twenty-four hours. In the first series of experiments, a sample of freshly distilled nitrobenzene was used, whereas that employed in the second series was obtained from the stock bottle containing nitrobenzene which had been exposed for some time to the air. There can

be no doubt that the latter sample contained a larger proportion of absorbed water, and, as might be expected, the conductivity values for the second series of solutions are greater than those for the first, corresponding with the greater solubility of the iodides in moist nitrobenzene. The two series of values are recorded in table V.

TABLE V.

Specific Conductivities of Saturated Solutions. $\kappa \cdot 10^4$.

	NH ₄ I.	NaI.	KI.	CsI.	NMe ₄ I.	RbI.
Series 1 ...	0.075	0.16	0.19	0.24	0.27	0.29
" 2 ...	0.11	—	0.26	0.39	—	0.40

These conductivity values are far too small to account for the difference in the sequence of the iodides which is found when the conductivities of 0.1 molar solutions of tri-iodide, prepared as described, are compared with those of solutions of penta-, hepta-, and ennea-iodide.

The regular decrease in the conductivity, which is met with in the case of all the metals and ammonium bases examined on passing from the penta- to the ennea-iodide, is an indication, either that the higher polyiodides of a given base are equally ionised, or that the ionisation varies with the proportion of iodine in the polyiodide in a way which is independent of the contained metal or ammonium radicle. In the case of solutions of potassium penta-, hepta-, and ennea-iodides, for which several concordant series of freezing-point determinations have been carried out, the observed freezing-point depressions are in good agreement with those calculated on the assumption that these polyiodides are electrolytically dissociated to the same extent as the tri-iodide. This agreement represents a strong argument in favour of the view that the higher polyiodides of potassium are equally ionised. If this is true in the case of potassium, the fact that the differences between the conductivity values of the penta-, hepta-, and ennea-iodides are independent of the cation makes it probable that the higher polyiodides of each alkali metal and ammonium radicle are ionised to the same extent. In these circumstances, the decrease in conductivity on passing from the penta- to the ennea-iodide corresponds with the diminution in the mobility of the polyiodide anion as its complexity increases.

If the higher polyiodides of all the different metals and ammonium bases were equally ionised, the differences in the conductivity values along the vertical series would, in a similar way, correspond with differences in the mobility of the various cations. Evidence has, however, been obtained that the degree of ionisation varies with the nature of the cation. The freezing points of solutions containing

different pentaiodides, the concentration in each case being represented by 0.1 mol. per litre of nitrobenzene, are not identical, as they should be if the ionisation were independent of the cation. The relative freezing-point depressions for potassium, ammonium, tetramethylammonium, and tetrapropylammonium pentaiodides were found to be 1.90, 1.83, 1.89, and 1.78 respectively. These numbers indicate that the higher polyiodides of ammonium and tetrapropylammonium are ionised to a smaller extent than those of potassium and tetramethylammonium.

In regard to the anomalous sequence of the cations when these are arranged according to the conductivities of solutions of the tri-iodide, the probable explanation is that the differences in the degree of ionisation of the various tri-iodides are not the same as the differences in the ionisation of the higher polyiodides. Although in the case of potassium the freezing-point depressions, calculated on the assumption that the higher polyiodides are ionised to the same extent as the tri-iodide, are in good agreement with the observed values, it is probable that this equality does not hold good generally. In support of this statement, it has been found that the ratio of the freezing-point depressions of the tri- and penta-iodides is considerably smaller for ammonium than it is for potassium. For this comparison, the ammonium polyiodides were chosen because of the considerable displacement of ammonia in the table of conductivity values when the tri-iodide and the higher polyiodide solutions are compared. The freezing-point data indicate that ammonium tri-iodide is ionised to a considerably smaller extent than the penta- (hepta- or enne-) iodide, and this is in agreement with the observed position of ammonium relatively to potassium in the table of conductivity data.

It is therefore probable that differences in the relative degrees of ionisation are the cause of the difference in order of the cations when the conductivities of the tri-iodide solutions, on the one hand, and of the higher polyiodides, on the other, are compared.

It has already been pointed out that, according to freezing-point and conductivity measurements, the degrees of ionisation of the iodide and tri-iodide of tetrapropylammonium differ very considerably. Further investigation has shown that the phenomenon is quite general, the solutions of the tri-iodides having a greater conductivity than the corresponding solutions of the iodides. On account of the very small solubility of the metallic iodides in pure nitrobenzene, small quantities of ethyl alcohol were added to this, in order that solutions of greater concentration might be obtained for the comparison of the conductivities of the iodides and tri-iodides. In table VI the composition of the solvent, the concentration of the solutions, and the molecular conductivities are recorded.

TABLE VI.

Composition of solvent.	Concentration of solution.	Solute.	Molecular conductivity.
Nitrobenzene	0.01 molar	NET_4I	26.1
"	"	NET_4I_3	27.6
"	0.01 "	NPr_4I	22.9
"	"	NPr_4I_3	25.3
Nitrobenzene + 5 volumes per cent. ethyl alcohol	0.005 "	NaI	16.0
" " " " "	"	NaI_3	30.0
" " " " "	0.005 "	NH_4I	16.3
" " " " "	"	NH_4I_3	31.1
" +10 " " " "	0.005 "	KI	25.2
" " " " "	"	KI_3	35.1
" " " " "	0.005 "	RbI	27.8
" " " " "	"	RbI_3	36.1
" " " " "	0.005 "	CsI	28.8
" " " " "	"	CsI_3	36.6

The pairs of numbers in the last column show clearly that the tri-iodide is in all cases a better conductor than the iodide at the same concentration. Since the tri-iodides are dissociated chemically to only a very small extent, the increased conductivity of the tri-iodide cannot be attributed to the presence of free iodine.

It will be observed that the differences between the conductivities of the tri-iodide and iodide are much larger for the alkali metals than for the substituted ammonium bases, and this is not attributable to the composite nature of the solvent used in the case of the alkali metal iodides. Experiments made to determine approximately the influence of the added ethyl alcohol on the conductivity differences indicate that, whilst the absolute conductivity values are increased on the addition of ethyl alcohol, the relative differences between the values for the tri-iodide and iodide are diminished.

The fact that tetrapropylammonium iodide is readily soluble in nitrobenzene has enabled us to examine more closely the change in the conductivity of solutions of this substance on the gradual addition of iodine. In table VII measurements made on 0.1 and 0.01 molar solutions are recorded. The two horizontal series of numbers represent respectively the number of mols. of iodine present in the solution per mol. of iodide and the molecular conductivities.

TABLE VII.

0.1 molar NPr_4I .							
Molar ratio $\text{I}_2/\text{NPr}_4\text{I}$...	0.0	0.25	0.5	0.75	1.0	1.5	2.0
Molecular conductivity.	13.06	13.79	14.81	16.31	17.33	16.44	15.91

0.01 molar NPr_4I .							
Molar ratio $\text{I}_2/\text{NPr}_4\text{I}$	0.0	0.25	0.5	0.75	1.0	8.0	
Molecular conductivity	22.9	23.3	23.55	24.0	25.0	25.2	

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In the case of the 0.1 molar solutions, the conductivity increases regularly with increasing concentration of iodine, a maximum being reached when the amount of iodine added corresponds with the complete conversion of the iodide into the tri-iodide; further addition of iodine results in a diminished conductivity corresponding with the data previously recorded in table IV. A similar initial increase, although of smaller magnitude, is also found in the case of the 0.01 molar solutions, while according to the last number in the series, an increase in the molar ratio I_2/NPr_4I from 1 to 8 has practically no effect on the conductivity of the solution. The slight increase actually observed is probably due to the fact that iodine itself, when dissolved in nitrobenzene, exhibits an appreciable conductivity. Several series of measurements have shown that the molar conductivity of iodine in nitrobenzene increases rapidly with the dilution of the solution, but the values obtained with samples of iodine and nitrobenzene, purified in different ways, diverge so widely that it is not possible to record definite values in this paper. It is obvious that the iodine resulting from the chemical dissociation of the higher polyiodides must be partly responsible for the conducting power of the polyiodide solutions, and, since the chemical dissociation of the polyiodides and the molecular conductivity of iodine both increase with dilution, the proportion of the total conductivity due to free iodine must increase as the dilution of the solution increases. The variation of the conductivity of the polyiodide solutions with the concentration will not be considered in this paper; for the present, it is sufficient to know that the significance of the conductivity values for 0.1 molar solutions recorded in table IV is not affected by the fact that iodine itself behaves as an electrolyte in nitrobenzene solution. In 0.01 molar solutions this property of iodine may possibly have some slight effect on the observed conductivities of the higher polyiodide solutions.

From the experimental data for tetrapropylammonium iodide solutions in tables IV and VII, and for the other iodide solutions in tables IV and VI, it is evident that the difference between the conducting properties of the iodide and the tri-iodide is much greater than, and opposite in sign to, the differences between successive members of the polyiodide series. Since it is highly probable that the tri-iodine ion has a smaller mobility than the simple iodine ion, the conductivity data lead to the conclusion that the simple iodides in nitrobenzene solution are ionised to a smaller extent than the tri-iodides and the higher polyiodides.

It may be noted that according to the numbers in table VI, the difference in the degree of ionisation of the iodide and the tri-iodide is much greater for the alkali metals and for ammonium than for

the tetra-substituted ammonium bases, whilst according to table VII the difference for tetrapropylammonium increases with increasing concentration.

The considerable difference in the degree of ionisation of the iodides, on the one hand, and of the polyiodides, on the other, stands in marked contrast with the corresponding ionisation phenomena in aqueous solution. It has been shown by one of us (Dawson, *Trans.*, 1901, 79, 238) that the ionisation "constants" of potassium iodide and potassium tri-iodide in aqueous solution are equal, and Burgess and Chapman (*Trans.*, 1904, 85, 1305), by two independent methods—determination of transport numbers and electrical conductivities—have confirmed this conclusion. Corresponding with this, the addition of iodine to an aqueous solution of potassium iodide results in a diminution of the electrical conductivity in consequence of the smaller mobility of the tri-iodine ion.

The facts indicate that the solvent exerts a very considerable influence on the relationship between the simple iodide and the polyiodides in so far as ionisation phenomena are concerned. It is not yet possible to account satisfactorily for the observed differences, although it would seem plausible to seek for an explanation in the combination of the solvent with the products of electrolytic dissociation.

Ionic Velocities.—It is obvious that if the share of the current carried by one of the ions of tetrapropylammonium iodide in nitrobenzene solution is known, the freezing-point and conductivity data for solutions of this substance may be utilised to deduce the ionic mobilities.

With this object in view, measurements of the transport number of the iodine ion were made by passing a known quantity of electricity through a 0.1 molar solution of tetrapropylammonium iodide contained in an apparatus consisting of a U-tube connected by a T-piece with a straight tube serving as anode chamber. The cathode consisted of a platinum disk, the anode of a small cylinder of metallic lithium. At the end of the experiment, an aqueous solution of the iodide in the anode tube was obtained by repeated extraction of the weighed nitrobenzene solution with water, any salt adhering to the anode being carefully removed in the process. The iodide was estimated by precipitation as silver iodide, the same method being used for the estimation of the iodide in the original solution. Two determinations gave 0.63 for the transport value of the iodine ion.

Denoting the velocities of the tetrapropylammonium and iodine ions by u and v_i , the following equations are obtained by combining the freezing-point, conductivity, and transport data:

$$13.06 = 0.35 (u + v_i)$$

$$0.63 = \frac{v_i}{u + v_i}.$$

From these, $u = 13.8$ and $v_t = 23.5$.

Combining these results with the freezing-point and conductivity data for the 0.1 molar solution of tetrapropylammonium tri-iodide, we obtain the equation:

$$17.38 = 0.68 (13.8 + v_{t3}),$$

which gives for the velocity of the tri-iodine ion, v_{t3} , the value 11.8. According to these numbers, the ratio of the velocities of the tri-iodine and iodine ions in nitrobenzene solution is 1:2.

From the mobility of the tri-iodine ion and the conductivity data for the polyiodide solutions recorded in table IV, it would be possible to deduce the mobilities of the various cations if the extent to which the polyiodides are ionised were accurately known. From the data for the potassium polyiodides, we obtain, on the assumption that the higher polyiodides are electrolytically dissociated to the same extent as the tri-iodide, the following values for the mobilities of the potassium and the polyiodide ions: $K = 16.6$, $I_3 = 9.7$, $I_7 = 9.2$, and $I_9 = 8.6$. On account of the considerable chemical dissociation of the hepta- and ennea-iodide, the values representing the mobilities of the hepta- and ennea-iodide anions can only be regarded as approximate.

Density Measurements.—Anticipating that the volume occupied by iodine in nitrobenzene solution might depend upon its condition, that is, whether it is present in the free state or combined in the form of a polyiodide (or polyiodide anion), determinations have been made of the molecular solution volumes of the various polyiodides of potassium.

An Ostwald pycnometer of about 20 c.c. capacity was employed in the density measurements, and a very large water-bath permitted the temperature of 18° to be maintained during the period of the experiments with a variation of less than $\pm 0.005^\circ$.

Iodine and iodide, in amounts corresponding with 0.01 mol. I_2 , KI_3 , KI_7 , and KI_9 , were weighed out into well-fitting stoppered bottles; 100 c.c. of nitrobenzene were then added, the exact amount of the solvent being determined by weighing. All weighings were reduced to a vacuum, and the molecular solution volumes calculated from the equation:

$$V = \left(\frac{s+g}{d} - \frac{s}{d'} \right) \frac{M}{g},$$

in which s and g denote the weights of solvent and solute, d and d' the densities of solution and solvent, and M the molar weight of the solute. In table VIII, V_1 and V_2 represent independently determined values of the molecular solution volume, V is the mean value, while the figures in the last column represent the differences

in the solution volume corresponding with a difference of one molecule of iodine in the composition of the solute.

TABLE VIII.

Solute.	V_1 (c.c.).	V_2 (c.c.).	V (c.c.).	Volume difference
				in c.c. per 1 mol. iodine.
I_2	67.0	68.1	67.5	
$KI + I_2$	101.6	101.9	101.8	
$KI + 2I_2$	165.0	166.1	165.5	63.7
$KI + 3I_2$	230.3	—	230.3	64.8
$KI + 4I_2$	296.4	—	296.4	66.1

The maximum difference between V_1 and V_2 , which represent values obtained by different observers, amounts to 1.1 c.c.; the possible error in the mean value may therefore be set down as about 0.5 c.c. An inspection of the figures in the last column shows that the volume occupied by the successive molecules of iodine in the series of polyiodides is less than the volume occupied by iodine in the free condition, but that it increases as the proportion of iodine in the polyiodides increases. Now the chemical dissociation of the triiodide is practically nil, and that of the penta-iodide only amounts to 2.5 per cent., so that the difference between the molecular solutions of these two substances represents the volume of iodine which is combined in the form of polyiodide. On the other hand, when we pass from the penta- to the hepta-iodide, and from this to the enne-iodide, a considerable proportion of the extra molecule of iodine involved is present in the free state, the proportion being greater for the second step than it is for the first. The fact that the differences of solution volume corresponding with one molecule of iodine increase with increasing complexity of the polyiodide, and tend towards the value for the molecular solution volume of free iodine in nitrobenzene, is therefore in agreement with the chemical dissociation phenomena as elucidated by the distribution experiments (*loc. cit.*).

With the object of ascertaining the volume of the molecule of iodine which is involved on passing from the iodide to the tri-iodide, the molecular solution volumes of tetrapropylammonium iodide and triiodide have been determined. This measurement is of course impossible in the case of potassium, by reason of the insolubility of the iodide. The data are recorded in table IX.

TABLE IX.

Solute.	V_1 (c.c.).	V_2 (c.c.).	V (c.c.).	Volume difference
				in c.c. per 1 mol. iodine.
NEt_4I	240.2	240.0	240.1	
NEt_4I_3	308.8	308.4	308.6	68.5

The value thus obtained for the molecular volume of the combined iodine in the tri-iodide is greater by 1 c.c. than the value found for uncombined iodine. This result, at first sight, appears to be contradictory to the observations made on potassium polyiodide solutions, but it must be remembered that the iodide and tri-iodide of tetrapropylammonium are ionised to very different extents. If the increased ionisation of the tri-iodide corresponds with an increase in volume, the high value obtained for the solution volume of the combined iodine in this case is at once accounted for. On account of this difference in the degree of electrolytic dissociation of the iodide and tri-iodide, the corresponding molecular solution volumes cannot be regarded as strictly comparable quantities. On the other hand, the polyiodides of potassium are equally ionised, and the differences in the molecular solution volumes of successive members of the series may be legitimately compared. These differences are in general agreement with the chemical dissociation phenomena exhibited by the polyiodides.

The following is a summary of the results obtained in this paper:

(1) The polyiodides of the alkali metals and the ammonium bases are electrolytically dissociated to a considerable extent in nitrobenzene solution.

(2) The penta-, hepta-, and enne-iodide of a particular metal or ammonium radicle are equally ionised, although there is evidence to show that the degree of ionisation depends on the cation.

(3) In certain cases, the tri-iodides are ionised to a somewhat smaller extent than the higher polyiodides.

In the case of the potassium compounds which were examined in greatest detail, there appears to be no difference between the tri- and higher poly-iodides.

(4) The simple iodides are ionised to a much smaller extent than the corresponding polyiodides.

(5) Values representing the mobilities of the polyiodide ions have been deduced.

(6) Measurements of the molecular solution volume of iodine indicate that this depends on whether the iodine is present in the free condition or combined in the form of a polyiodide.

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PHYSICAL CHEMISTRY LABORATORY,
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LEEDS.

(CV.)—*Meteloidine: A New Solanaceous Alkaloid.*

By FRANK LEE PYMAN and WILLIAM COLEBROOK REYNOLDS.

Datura Meteloides is a small shrub closely resembling *D. Metel*, as its name implies. Botanically, it differs from the latter in several respects (Dunal in De Candolle, *Prod.*, xiii, 1, 554), having for instance smaller leaves and a pink flower, whereas the flower of *D. Metel* is white.

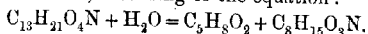
Schmidt (*Arch. Pharm.*, 1905, 243, 303) and Kircher (*Arch. Pharm.*, 1905, 243, 309) have shown that *D. Metel* contains about 0·5 per cent. of alkaloids, consisting mainly of hyoscyne with a small amount of atropine and hyoscyamine. *Datura Meteloides*, however, does not appear to have been investigated chemically hitherto. It has now been found to contain 0·4 per cent. of total alkaloids, from which 0·13 per cent. of hyoscyne, 0·03 per cent. of atropine, and 0·07 per cent. of a new alkaloid have been obtained; the latter has been designated *meteloidine* with reference to its source.

Meteloidine is a crystalline base of the empirical formula $C_{13}H_{21}O_4N$, and forms salts which crystallise readily; the *hydrobromide*,



aureichloride, $C_{13}H_{21}O_4N \cdot HAuCl_4 \cdot \frac{1}{2}H_2O$, and *picrate* have been prepared. It is optically inactive, and has no marked physiological action.

When boiled with aqueous barium hydroxide, meteloidine is hydrolysed with formation of tiglic acid and a new base, which has been designated *teloidine*, according to the equation:



The formation of tiglic acid on hydrolysis of an alkaloid has been observed on only one previous occasion, when Wright and Luff (*Trans.*, 1878, 33, 347) obtained it from cevadine, one of the alkaloids of *Veratrum Sabadilla*.

The basic hydrolytic product, teloidine, crystallises from moist acetone with one molecule of water of crystallisation. It forms easily crystallisable salts, of which the *hydrochloride*, $C_8H_{15}O_3N \cdot HCl$, *hydrobromide*, $C_8H_{15}O_3N \cdot HBr$, and *aureichloride*, $C_8H_{15}O_3N \cdot HAuCl_4 \cdot \frac{1}{2}H_2O$, have been characterised. Lack of material has prevented further investigation of this substance, but it may be pointed out that it has certain properties in common with tropine and oscine, and may conceivably be closely related to these bases. The three substances are all strongly basic, and each contains eight carbon atoms and a hydroxyl group in the molecule; they are all very soluble in water and alcohol, and are left as oily residues on the evaporation of these solvents; concentrated aqueous solutions of their salts give no pre-

precipitate with picric acid. It is interesting to note also that acyl derivatives of the three bases are found in *D. Meteloides*, atropine or tropyltropine, hyoscyne or tropanyloseine, and meteloidine or tiglylteloidine.

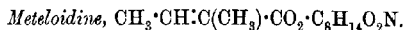
On the other hand, teloidine differs from tropine and oscine in that it cannot be distilled, and concentrated aqueous solutions of its salts do not give a precipitate with mercuric-potassium iodide solution.

EXPERIMENTAL.

Isolation of Meteloidine from Datura Meteloides.

The powdered herb was percolated with 95 per cent. alcohol, and the extract concentrated to a semi-solid mass, from which the alkaloids were removed by stirring with 1 per cent. aqueous hydrochloric acid. The aqueous liquor was rendered alkaline with ammonia, and shaken out with chloroform, and the latter fractionally extracted with successive quantities of dilute hydrobromic acid. The extracts were concentrated separately, and allowed to crystallise. Most, if not all, of the meteloidine was contained in the first fraction, and crystallised out on cooling as the hydrobromide. From the mother liquor of this and the later hydrobromic acid extracts, hyoscyne and a mixture of hyoscyamine and atropine were separated in the usual manner.

Out of a yield of 0.40 per cent. of total alkaloids, 0.07 per cent. of meteloidine, 0.12 per cent. of hyoscyne, and 0.03 per cent. of atropine were obtained.



The base was prepared by adding sodium carbonate to an aqueous solution of the hydrobromide, extracting with chloroform, and crystallising the chloroform residue from benzene. It separated in tabular needles, which melted at 141—142° (corr.). It is readily soluble in alcohol, acetone, or chloroform, and somewhat sparingly so in water, ether, ethyl acetate, or benzene:

0.1162 gave 0.2610 CO_2 and 0.0874 H_2O . $\text{C} = 61.2$; $\text{H} = 8.4$.

0.0702 „ 3.4 c.c. N_2 at 19° and 764 mm. $\text{N} = 5.6$.

$\text{C}_{13}\text{H}_{21}\text{O}_4\text{N}$ requires $\text{C} = 61.1$; $\text{H} = 8.3$; $\text{N} = 5.5$ per cent.

The *hydrobromide* crystallised from water in hard clusters of chisel-shaped needles. It contains two molecules of water of crystallisation, and is readily soluble in water and absolute alcohol. After drying first at 40° and then at 100°, it melts and colours at 250° (corr.). It is optically inactive:

0.1858, air-dried, gave 0.2854 CO_2 and 0.1186 H_2O . $\text{C} = 41.9$; $\text{H} = 7.2$.

0.2726 „ „ lost 0.0258 at 100°. $\text{H}_2\text{O} = 9.5$.

0.1444, dried at 100°, gave 0.0804 AgBr. Br = 23.7.
 $C_{13}H_{21}O_4N.HBr.2H_2O$ requires C = 41.9; H = 7.1; H_2O = 9.7 per cent.
 $C_{13}H_{21}O_4N.HBr$ requires Br = 23.8 per cent.

The *aurichloride* crystallised on adding auric chloride to a solution of the hydrochloride. After recrystallisation from dilute alcohol, it formed short, yellow needles, which melted at 149–150°. It contains one-half of a molecular proportion of water of crystallisation, and is sparingly soluble in water, but readily so in alcohol:

0.0937, air-dried, lost 0.0017 at 100°. H_2O = 1.8.

0.0940, dried at 100°, gave 0.0315 Au. Au = 33.5.

$C_{13}H_{21}O_4N.HAuCl_4.1\frac{1}{2}H_2O$ requires H_2O = 1.5 per cent.

$C_{13}H_{21}O_4N.HAuCl_4$ requires Au = 33.1 per cent.

The *picrate* separated in yellow, crystalline nodules on adding picric acid to a solution of the hydrobromide. After recrystallisation from alcohol, it formed yellow, hexagonal plates, which melted at 177–180° (corr.). It is sparingly soluble in water and cold alcohol.

Hydrolysis of Meteloidine by Barium Hydroxide.

Formation of Tiglic Acid and Teloidine.

Twelve grams of meteloidine hydrobromide were converted into the free base, and this was dissolved in a warm solution of 10.2 grams of crystallised barium hydroxide in 150 c.c. of water and boiled for one hour. After cooling, the solution was extracted with chloroform, which removed 0.3 gram of unchanged meteloidine, boiled, acidified with 12 c.c. of 50 per cent. aqueous sulphuric acid, filtered from precipitated barium sulphate, and completely extracted with ether. The ethereal extract was dried, and on distillation gave 2.8 grams of a crystalline acid; on recrystallisation from water, it separated in long, flat needles, which melted at 64–64.5° (corr.), and proved to be tiglic acid:

0.1456 gave 0.3174 CO_2 and 0.1050 H_2O . C = 59.5; H = 8.1.

$C_8H_8O_2$ requires C = 60.0; H = 8.1 per cent.

For further proof of identity, the acid was converted into its dibromide, which separated from light petroleum in long, white needles, which melted at 87–88° (corr.).

The yield of tiglic acid, based on the amount of meteloidine actually hydrolysed, is 91 per cent. of the theoretical.

The acid liquor remaining after the ether extraction was digested on the water-bath with barium carbonate and filtered, when it was found to be faintly alkaline. After neutralisation with a drop of dilute sulphuric acid, and evaporation to low bulk on the water-bath, it began to crystallise, and was then completely dried,

first on the water-bath, then in a vacuum desiccator over sulphuric acid, and gave 6.2 grams of a dry, pale pink, crystalline powder. This proved to be teloidine hydrochloride mixed with a small proportion of sulphate, and the amount obtained represents 96 per cent. of the theoretical. The occurrence of the hydrochloric acid can only have been due to the decomposition of the chloroform used earlier in the process.

Teloidine, $C_8H_{15}O_3N$.

The free base cannot be extracted from an aqueous solution by shaking with the usual organic solvents. It was isolated in the following manner: The hydrochloride was dissolved in its own weight of water, mixed with a strong aqueous solution of one molecular proportion of potassium hydroxide, and diluted with absolute alcohol. After filtering from precipitated potassium chloride, the solution was evaporated to dryness under diminished pressure, and the residue again extracted with absolute alcohol, filtered, and evaporated to dryness, when the base was obtained as a viscid oil. This was dissolved by covering with about 10 volumes of boiling acetone and adding water drop by drop until nearly all had passed into solution. After filtering hot, the solution was allowed to stand, when the base separated in chisel-shaped needles, which contained one molecule of water of crystallisation. After drying at 120° , it melted at $168-169^\circ$ (corr.):

0.1226, air-dried, gave 0.2244 CO_2 and 0.0990 H_2O . $C = 49.9$; $H = 9.1$.

0.1382 ,, ,, 8.6 c.c. N_2 at 21° and 771 mm. $N = 7.2$.

0.2194 ,, lost 0.0199 at 120° . $H_2O = 9.1$.

$C_8H_{15}O_3N, H_2O$ requires $C = 50.2$; $H = 9.0$; $N = 7.3$; $H_2O = 9.4$ per cent.

0.1438, dried at 120° , gave 0.2906 CO_2 and 0.1142 H_2O . $C = 55.1$; $H = 8.9$.

$C_8H_{15}O_3N$ requires $C = 55.4$; $H = 8.7$ per cent.

Teloidine is not deliquescent, nor does the anhydrous base take up water of crystallisation from moist air. It is very easily soluble in water or alcohol, and is left as an oil on the evaporation of these solvents; it is sparingly soluble in the other usual organic solvents when cold. It gives no precipitate with picric acid or mercuric potassium iodide even in concentrated solution. It cannot be distilled, but suffers decomposition.

The *hydrochloride* is obtained almost pure by the above method: after recrystallisation from absolute alcohol, it separates in well defined, monoclinic prisms with a macrodome, and does not melt at 300° . It is anhydrous, and is readily soluble in water, but sparingly so in boiling absolute alcohol:

0.1576 gave 0.2634 CO_2 and 0.1102 H_2O . $C = 45.6$; $H = 7.8$.

0.1678 gave 9.6 c.c. N_2 at 21° and 766 mm. $N = 6.6$.

0.1803 " 0.1221 AgCl. $Cl = 16.7$.

$C_8H_{15}O_3N$, HCl requires $C = 45.8$; $H = 7.7$; $N = 6.7$; $Cl = 16.9$ per cent.

The *hydrobromide* crystallised from absolute alcohol in hexagonal plates and needles, which melted and decomposed at 295° (corr.). It is anhydrous, and is readily soluble in water, but sparingly so in absolute alcohol:

0.1694 gave 0.2342 CO_2 and 0.0954 H_2O . $C = 37.7$; $H = 6.3$.

0.1776 " 0.1301 AgBr. $Br = 31.2$.

$C_8H_{15}O_3N$, HBr requires $C = 37.8$; $H = 6.4$; $Br = 31.5$ per cent.

The *aurochloride* crystallised out on adding a concentrated solution of auric chloride to a strong solution of the hydrochloride. After recrystallisation from water, it separated in yellow, hexagonal plates, which melted and decomposed at 225° (corr.). It contains one-half of a molecular proportion of water of crystallisation, and is fairly easily soluble in water:

0.1919, air-dried, lost 0.0032 at 100° . $H_2O = 1.7$.

0.1887, dried at 100° , gave 0.0721 Au. $Au = 38.2$.

$C_8H_{15}O_3N$, $HAuCl_4 \cdot \frac{1}{2}H_2O$ requires $H_2O = 1.7$ per cent.

$C_8H_{15}O_3N$, $HAuCl_4$ requires $Au = 38.4$ per cent.

With the object of determining the number of hydroxy-groups in the molecule, teloidine has been subjected to the action of boiling acetic anhydride and benzoyl chloride in aqueous sodium hydroxide, but no crystalline derivatives were obtained from the small quantities employed.

It is proposed to investigate further the constitution of meteloidine when more material is available.

THE WELLCOME CHEMICAL WORKS,
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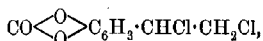
(CVI).—*The Action of Phosphorus Pentachloride on the Methylene Ethers of Catechol Derivatives. Part III. The Cyclic Carbonates of Dichloro-ethyl- and -propyl-catechol.*

By GEORGE BARGER.

SOME years ago, in conjunction with Dr. H. A. D. Jowett, the author made an attempt to convert $\alpha\beta$ -dibromo- α -3 : 4-methylenedioxyphenylethane, $CH_2 \begin{smallmatrix} \diagup O \\ \diagdown O \end{smallmatrix} C_6H_3 \cdot CHBr \cdot CH_2Br$ (TRANS, 1905, 87, 967), into the

corresponding catechol derivative by Fittig and Remsen's reaction. The only product which could be isolated was a small quantity of a dibromohydrin, $\text{CH}_2\text{:O}_2\text{:C}_6\text{H}_3\text{Br}\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\text{Br}$, formed by the action of bromine which had been liberated by the phosphorus pentachloride. When the subject was again taken up more recently, an attempt was made to use thionyl chloride instead of phosphorus pentachloride, for, as the author has shown (*Trans.*, 1908, 93, 563), the former reagent converts catechol methylene ethers directly into cyclic carbonates. In the present case, however, the reaction proceeded in an anomalous manner, and instead of the desired substance, a thionaphthen derivative was formed, as described in the next paper (p. 2086). After this failure, recourse was again had to phosphorus pentachloride, and, in order to exclude the secondary action of liberated bromine, the dibromide previously employed was replaced by the corre-

sponding dichloride. This substance, $\text{CH}_2\text{<O>C}_6\text{H}_3\cdot\text{CHCl}\cdot\text{CH}_2\text{Cl}$, was readily prepared from the corresponding styrene, but as it could only be obtained as a viscid oil, it was found convenient to convert it into the crystalline chlorohydrin, $\text{CH}_2\text{<O>C}_6\text{H}_3\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\text{Cl}$, which is easily obtained pure. On submitting the chlorohydrin to the action of three molecular proportions of phosphorus pentachloride, it is reconverted into the dichloride and then transformed into a dichloromethylene derivative, $\text{CCl}_2\text{<O>C}_6\text{H}_3\cdot\text{CHCl}\cdot\text{CH}_2\text{Cl}$. The latter substance not only distills without decomposition, but, unlike the known examples of this unstable type, it can be obtained crystalline. By treatment with anhydrous formic acid, the cyclic carbonate,

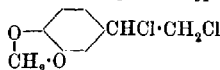


results, which is a derivative of 3:4-dihydroxy-1-ethylbenzene, prepared by Delange (*Compt. rend.*, 1904, 138, 423). The preparation of the corresponding dibromide, $\text{CO<O>C}_6\text{H}_3\cdot\text{CHBr}\cdot\text{CH}_2\text{Br}$, was recently attempted unsuccessfully by Pauly and Neukam (*Ber.*, 1907, 40, 3488) by the addition of bromine to vinylcatechol carbonate. From the easily obtainable *isosafrole* dichloride, by successive treatment with phosphorus pentachloride and formic acid, the cyclic carbonate, $\text{CO<O>C}_6\text{H}_3\cdot\text{CHCl}\cdot\text{CHCl}\cdot\text{CH}_3$, was prepared; it closely resembling its lower homologue. By boiling water, both carbonates are readily hydrolysed to catechol compounds, but, in addition, the α -chlorine atom is removed. So far it has not been possible to isolate the chlorohydrin, $\text{C}_6\text{H}_3(\text{HO})_2\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\text{Cl}$; it would appear that,

under the conditions hitherto employed, hydrogen chloride is eliminated and a chlorovinyl derivative is formed. Further experiments are, however, in progress.

EXPERIMENTAL.

αβ-Dichloro-α-3 : 4-methylenedioxyphenylethane,



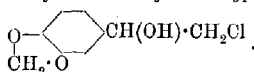
To 15 grams of 3 : 4-methylenedioxytoluene (Klages, *Ber.*, 1903, 36, 3595), dissolved in carbon tetrachloride, a solution of chlorine in the same solvent was added until a sample no longer decolorised a dilute solution of bromine. On distillation, 16 grams of the crude dichloride passed over at 160—180°/16 mm. It constituted a viscid liquid, which could not be crystallised. As it could not readily be purified by fractional distillation, it was converted into the chlorohydrin (described below), and this was reconverted into the dichloride by heating on the water-bath with thionyl chloride. The regenerated dichloride then boiled at 159—160°/12 mm., and was not quite pure, as shown by the analysis :

0.1475 gave 0.1837 AgCl. Cl = 30.8.

$C_9H_8O_2Cl_2$ requires Cl = 32.4 per cent.

There would appear to be a tendency to lose hydrogen chloride.

β-Chloro-α-hydroxy-α-3 : 4-methylenedioxyphenylethane,



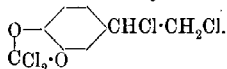
This chlorohydrin was prepared by leaving the crude dichloride (above) for one day, dissolved in a mixture of acetone and water (compare Auwers and Miller, *Ber.*, 1902, 35, 114), or, preferably, by heating the solution in aqueous acetone with powdered marble, on the water-bath under a reflux condenser. After removal of the acetone and water by distillation, the partly solid residue was pressed on a porous plate and crystallised from a mixture of light petroleum and benzene, when it formed long needles melting at 95° and boiling at 169—170°/7 mm. :

0.1398 gave 0.2756 CO_2 and 0.0560 H_2O . C = 53.8 ; H = 4.4.

0.2023 „ 0.1474 AgCl. Cl = 18.0

$C_9H_9O_3Cl$ requires C = 53.9 ; H = 4.5 ; Cl = 17.7 per cent.

The yield of this substance and the preceding one was much inferior to that of the corresponding dibromide and bromohydrin.

αβ-Dichloro-α-3 : 4-dichloromethylenedioxyphenylethane,

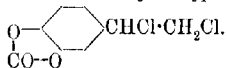
The dichloride and chlorohydrin were heated respectively with two and three molecular proportions of phosphorus pentachloride to 150—170° for four hours. The flask was provided with a Young's still-head, so as to allow the phosphorus trichloride and oxychloride to distil over, but to retain the pentachloride. At the end of the reaction, a small quantity of phosphorus pentachloride was sublimed out of the flask under diminished pressure, and the contents were then distilled under a pressure not exceeding 10 mm. Under suitable conditions, the yield is almost quantitative; thus, for instance, 5 grams of the chlorohydrin, heated with 15.7 grams of phosphorus pentachloride to 150° for six hours, yielded 6.5 grams of a distillate boiling at 170—174°/6 mm. (the theoretical yield is 7.1 grams). On one occasion, during a severe frost, the distillate, which was left overnight in the receiver, solidified; it was pressed on a porous tile, and could then be crystallised from light petroleum and benzene, yielding prisms which melted at 56°. Later crystallisation could never be produced spontaneously, but only by inoculation :

0.1758 gave 0.2535 CO_2 and 0.0372 H_2O . $\text{C} = 39.3$; $\text{H} = 2.3$.

0.1632 „ 0.3206 AgCl . $\text{Cl} = 48.6$.

$\text{C}_9\text{H}_6\text{O}_2\text{Cl}_4$ requires $\text{C} = 39.6$; $\text{H} = 2.1$; $\text{Cl} = 49.3$ per cent.

The crystals fume in moist air and gradually liquefy ; when dissolved in acetone, the substance reacts violently with water, the temperature of the acetone being raised to the boiling point. With anhydrous formic acid fumes of hydrogen chloride are rapidly evolved without much heat, and the cyclic carbonate is formed.

αβ-Dichloro-α-3 : 4-carbonyldioxyphenylethane,

Anhydrous formic (or acetic) acid is added carefully in small quantities to the dichloromethylene derivative described above, and the excess is removed by distillation under diminished pressure. This completes the reaction, and the carbonate can then be distilled. It boils at 180—182°/8 mm.,* the yield being quantitative.

A fraction b. p. 182°/8 mm. was analysed :

* At, or about, this pressure the cyclic catechol carbonates generally boil 10° higher than the dichloromethylene derivatives, from which they are obtained.

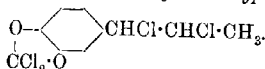
0.1573 gave 0.2624 CO_2 and 0.0390 H_2O . $\text{C} = 46.2$; $\text{H} = 2.7$.

0.1842 „ 0.2275 AgCl . $\text{Cl} = 30.5$.

$\text{C}_{10}\text{H}_6\text{O}_3\text{Cl}_2$ requires $\text{C} = 46.4$; $\text{H} = 2.6$; $\text{Cl} = 30.5$ per cent.

Although the analysis proves the substance to be quite pure, it could not be crystallised. At the room temperature, it is a very viscid oil, readily soluble in all ordinary organic solvents, except light petroleum. When boiled with water, it dissolves gradually, being converted into a catechol derivative, but at the same time a large amount of a dark brown resin is formed.

αβ-Dichloro-α-3:4-dichloromethylenedioxyphenylpropane,



This substance can readily be prepared in large quantities from *isosafrole* dichloride by the method employed for the ethane derivative. Thus, for example, 47 grams of *isosafrole* dichloride (b. p. 157—161°/8 mm.) yielded, after heating with two molecules of phosphorus pentachloride, a distillate which, under a pressure of 8 mm., boiled between 173° and 180°, mostly at 175°. A slight residue was left in the flask.

The fraction b. p. 174.5—175.5°/8 mm. was analysed:

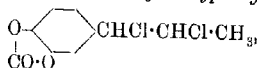
0.2184 gave 0.3184 CO_2 and 0.0617 H_2O . $\text{C} = 39.7$; $\text{H} = 3.1$.

0.1680 „ 0.3212 AgCl . $\text{Cl} = 47.3$.

$\text{C}_{10}\text{H}_8\text{O}_2\text{Cl}_4$ requires $\text{C} = 39.7$; $\text{H} = 2.6$; $\text{Cl} = 47.0$ per cent.

The fractions boiling at a higher temperature were contaminated with a substance containing more chlorine.

αβ-Dichloro-α-3:4-carboxyldioxyphenylpropane,



After treatment of the previous substance with formic acid, the carbonate is obtained as a viscid oil, b. p. 186—189°/8 mm., closely resembling the lower homologue:

0.1760 gave 0.3111 CO_2 and 0.0522 H_2O . $\text{C} = 48.2$; $\text{H} = 3.3$.

0.2240 „ 0.2687 AgCl . $\text{Cl} = 28.7$.

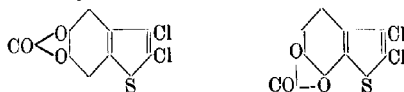
$\text{C}_{10}\text{H}_8\text{O}_3\text{Cl}_2$ requires $\text{C} = 48.6$; $\text{H} = 3.2$; $\text{Cl} = 28.7$ per cent.

THE WELLCOME PHYSIOLOGICAL RESEARCH LABORATORIES,
BROCKWELL HALL, HERNE HILL, LONDON, S.E.

CCVII.—*The Synthesis of Thionaphthen Derivatives from Styrenes and Thionyl Chloride.*

By GEORGE BARGER and ARTHUR JAMES EWINS.

In an attempt to convert 3 : 4-methylenedioxyphenylmethylcarbinol, $\text{CH}_3\text{O}_2\text{C}_6\text{H}_3\text{CH}(\text{OH})\text{CH}_3$, into the corresponding cyclic carbonate by means of thionyl chloride (compare the preceding paper and Traus, 1908, 93, 563), we found that the reaction proceeds in an unexpected manner; the resulting substance is, indeed, a cyclic carbonate, but it contains sulphur and has the composition $\text{C}_9\text{H}_2\text{O}_3\text{Cl}_2\text{S}$. The same compound was produced when $\alpha\beta$ -dibromo-3 : 4-methylenedioxy-styrene was acted on by thionyl chloride, the bromine being eliminated. Since it contains only two hydrogen atoms, the new substance must possess a condensed ring; it is most probably a thionaphthen derivative, and may be represented by one of the following formulæ:



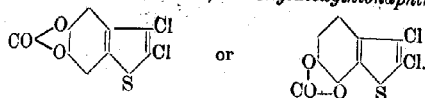
In order to determine whether the reaction is a general one, we submitted derivatives of other styrenes to the action of thionyl chloride. Although organic sulphur derivatives were formed in each case, we were not always able to isolate them from the mixture produced. Thus, from ordinary styrene, we could only obtain a pure substance after we had employed conditions favourable to complete chlorination, the resulting compound being *hexachlorothionaphthen*, $\text{C}_8\text{Cl}_6\text{S}$.

The substances described below do not give the indophenine reaction, but some of them, containing relatively little chlorine, gave, after reduction, a green or bluish-green coloration with isatin and sulphuric acid; the typical blue coloration was never obtained.

In all cases a large excess of thionyl chloride was used, and was heated with the styrene derivative in a sealed tube (to $180\text{--}270^\circ$). An attempt to carry out the reaction at a low temperature with aluminium chloride and thionyl chloride or sulphur dioxide was unsuccessful. Dibromostyrene was generally employed, not only because it was most readily obtainable, but because it alone was found to yield a crystalline product when the dichloro-derivative did not, even in the presence of iodine. For this superior reactivity of the bromo-compounds we cannot offer an explanation. The yield of pure thionaphthen derivatives was generally small, except in the case of the catechol derivative first investigated, when it amounted to 70 per cent. of the theoretical.

EXPERIMENTAL

1:2-Dichloro-4:5(or 5:6)-carbonyldioxythionaphthen,



2.2 Grams of 3:4-methylenedioxyphenylmethylcarbinol were heated with 13 grams of thionyl chloride for five hours in a sealed tube at 170° . On opening the tube there was found to be a great pressure; the contents were a reddish-brown liquid and a crystalline solid (the thionaphthen). The latter was collected and recrystallised from benzene; the yield was 45 per cent. of the theoretical.

One gram of $\alpha\beta$ -dibromo-3:4-methylenedioxy-styrene, heated with 5 grams of thionyl chloride to $250-260^{\circ}$ for seven hours, yielded 0.6 gram of the same substance or 70 per cent. of the theoretical. After repeated crystallisation from benzene, the substance from either source formed white prisms melting at 198° :

0.1668 gave 0.2514 CO_2 and 0.0124 H_2O . $\text{C} = 41.1$; $\text{H} = 0.8$.

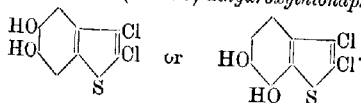
0.1151 „ 0.1251 AgCl . $\text{Cl} = 26.9$.

0.2472 „ 0.2150 BaSO_4 . $\text{S} = 11.9$.

0.3798, in 11.74 naphthalene, gave $\Delta = -0.87^{\circ}$. $\text{M.W.} = 257$.
 $\text{C}_9\text{H}_5\text{O}_3\text{Cl}_2\text{S}$ requires $\text{C} = 41.4$; $\text{H} = 0.8$; $\text{Cl} = 27.2$; $\text{S} = 12.2$ per cent.;
 $\text{M.W.} = 261$.

In addition to the secondary alcohol and the dibromo-derivative already mentioned, the following substances also yielded this thionaphthen derivative on heating with thionyl chloride: the ether, $(\text{CH}_2\text{O}_2\text{C}_6\text{H}_3\text{CHMe})_2\text{O}$; the dichloride, $\text{CH}_2\text{O}_2\text{C}_6\text{H}_3\text{CHClCH}_2\text{Cl}$, and the corresponding chloro- and bromo-hydrins. Since the side-chain in all these cases is readily chlorinated, the chlorine atoms in the resulting thionaphthen derivative are in all probability in positions 1 and 2. The para-hydroxyl group must be in position 5, and be meta-hydroxyl in either 4 or 6; that the substance is a cyclic carbonate is proved below.

1:2-Dichloro-4:5(or 5:6)-dihydroxythionaphthen,



The previously-described substance, $\text{C}_9\text{H}_5\text{O}_3\text{Cl}_2\text{S}$, was hydrolysed by gently warming it in an atmosphere of hydrogen with 50 per cent. aqueous pyridine; carbon dioxide was evolved. The yellow solution

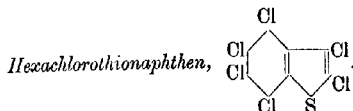
was evaporated in a vacuum over sulphuric acid, the residue was dissolved in water, and the aqueous solution extracted with ether. After washing with dilute sulphuric acid, drying, and evaporating the ethereal solution, the remaining solid was dissolved in hot benzene, from which it separated on cooling as a white, gelatinous mass melting and decomposing at 148° :

0.1482 gave 0.1848 AgCl and 0.1448 BaSO₄. Cl = 30.8; S = 13.4.
 $C_8H_4O_2Cl_2S$ requires Cl = 30.2; S = 13.6 per cent.

The dichlorodihydroxythionaphthen thus prepared is soluble in water and in most organic solvents. Its aqueous solution yields with ferric chloride a bluish-green coloration, differing somewhat from the pure green obtained with simple catechol derivatives.

The dibenzoyl derivative was prepared by Einhorn's method, and yielded, on crystallisation from a mixture of alcohol and benzene, long, slender needles melting at 185° :

0.1503 gave 0.0993 AgCl. Cl = 16.3.
 $C_{22}H_{12}O_4Cl_2S$ requires Cl = 16.0 per cent.



Two grams of $\alpha\beta$ -dibromostyrene were heated with 5 c.c. of thionyl chloride and 2 c.c. of sulphuryl chloride to 270° for thirteen hours. On cooling, the tube contained a red liquid in which crystals were suspended. The latter were recrystallised from light petroleum (b. p. $70-100^{\circ}$); they formed long, silky, white needles melting at 158° :

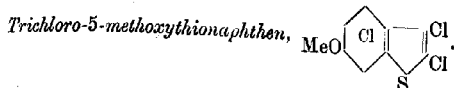
0.0959 gave 0.0977 CO₂ and 0.0005 H₂O. C = 27.8; H = 0.06.
 0.1640 „ 0.4189 AgCl and 0.1188 BaSO₄. Cl = 63.2; S = 9.9.
 C_8Cl_6S requires C = 28.1; Cl = 62.5; S = 9.4 per cent.

In a previous experiment, when the sulphuryl chloride was omitted and the temperature maintained at $215-240^{\circ}$ for ten hours, the same crystalline hexachlorothionaphthen was obtained, but the analysis showed it to be impure. Subsequently, sulphuryl chloride and a higher temperature were used to ensure complete chlorination.

The (crude) reduction product of hexachlorothionaphthen with hydriodic acid and phosphorus gave a reddish-purple coloration with isatin and sulphuric acid, changing to green on standing.

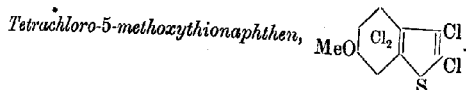
When heated with thionyl chloride to 210° for four to five hours, $\alpha\beta$ -dichlorostyrene furnished a distillate (b. p. $150-175^{\circ}/16$ mm.) containing both chlorine and sulphur, but no crystalline product

could be obtained, nor was any obtainable from phenylmethylcarbinol, even after heating to 240° for twenty hours. The distillate in the latter case gave a brilliant red coloration with isatin and sulphuric acid, and, after distillation with zinc dust, the same reagents produced a green coloration.



By heating 1.5 grams of *p*-methoxyphenylmethylcarbinol (from anisaldehyde) with 12 grams of thionyl chloride for three hours to 200° , and distilling under diminished pressure, a viscid liquid was obtained (b. p. $195-215^{\circ}/12$ mm.), which yielded a small quantity of a substance crystallising from light petroleum in white needles and melting at 153° :

0.0747 gave 0.1152 AgCl and 0.0649 BaSO₄. Cl = 38.2; S = 12.1.
 $C_9H_5OCl_3S$ requires Cl = 39.8; S = 12.0 per cent.



By heating 1.5 grams of *p*-methoxyphenylmethylcarbinol with 12 grams of thionyl chloride to 180° for eight hours, and removal of the excess of thionyl chloride on the water-bath, 3.6 grams of a distillate were collected, b. p. $200-225^{\circ}/12$ mm.

On dissolving in hot light petroleum (b. p. $70-100^{\circ}$) and cooling, 1.35 gram of silky, white needles, m. p. $109-111^{\circ}$, separated.

The melting point was unchanged by recrystallisation:

0.1536 gave 0.2966 AgCl and 0.1265 BaSO₄. Cl = 47.7; S = 11.3.
 $C_9H_4OCl_4S$ requires Cl = 46.8; S = 10.6 per cent.

As the analyses show, neither this nor the previous substance was obtained quite pure; they are formed simultaneously, and the small quantities at our disposal did not enable us to effect a complete separation. In either case the methoxy-group must be in position 5; but of the chlorine atoms in the benzene ring is doubtful.

Action of Thionyl Chloride on isoSafrole Dibromide.

isoSafrole dibromide reacts less readily with thionyl chloride than the lower homologue ($\alpha\beta$ -dibromomethylenedioxystyrene), but, nevertheless, yielded a small quantity of a thionaphthen derivative. From safrole dichloride, however, this derivative could not be obtained;

thus again illustrating the greater reactivity of the bromo-compound as compared with the chloro-compound.

Dichlorocarbonyldiozymethylthionaphthen was obtained by heating 3 grams of isosafrole dibromide with 13 grams of thionyl chloride to 220° for seven hours. On opening the tube, a few crystals were found to have separated, and, after cooling to -80°, 0.15 gram was collected. On recrystallisation from light petroleum (b. p. 70—100°), the substance melted at 180° :

0.1012 gave 0.1049 AgCl and 0.0879 BaSO₄. Cl = 25.6 ; S = 11.9.

C₁₀H₄O₃Cl₂S requires Cl = 25.8 ; S = 11.6 per cent.

The constitution is doubtful, but the methyl group is most probably in position 1.

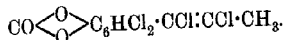
In addition to the above, a substance was obtained, which crystallised from light petroleum, melted at 167°, and did not contain sulphur :

0.1288 gave 0.1793 CO₂ and 0.0115 H₂O. C = 37.9 ; H = 1.0.

0.1689 „ 0.3097 AgCl. Cl = 45.3.

C₁₀H₄O₃Cl₄ requires C = 38.2 ; H = 1.2 ; Cl = 45.2 per cent.

This substance is most probably represented by the formula



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CCVIII.—*Organic Derivatives of Silicon. Part VIII.* *The Resolution of dl-Sulphobenzylethylisobutylsilicyl* *Oxide and the Properties of the Optically Active* *Acids.*

By BERNARD DUNSTAN WILKINSON LUFF (1851 Exhibition Scholar)
and FREDERIC STANLEY KIPPING.

OF the many optically active bases which were tried in the experiments on the resolution of *dl*-sulphobenzylethylpropylsilicyl oxide, only one, namely, methylhydrindamine, proved to be of any use; as it was perhaps to be expected that this same base would prove serviceable for the resolution of *dl*-sulphobenzylethylisobutylsilicyl oxide (Part VII), the pure sodium salt of the *dl*-acid, prepared from the pure *l*-menthylamine salt (this vol., p. 2010), was directly precipitated with excess of *l*-methylhydrindamine hydrochloride in aqueous solu-

tion. The oily product, which slowly solidified, was then fractionally crystallised from aqueous methyl alcohol. After a series of operations, we succeeded in isolating a colourless, crystalline salt melting at $207-209^{\circ}$, which proved to be the *l*-methylhydrindamine salt of *l*-sulphobenzylethylisobutylsilicyl oxide. The aqueous-alcoholic mother liquors from this compound deposited the optically impure isomeric salt of the *d*-acid as an oil.

Direct evidence that the *dl*-silicon compound had been resolved was obtained by treating the pure salt of the *l*-acid with sodium carbonate, expelling the methylhydrindamine, and examining the solution of the sodium salt in the polarimeter. The specific rotation calculated for the sodium salt was found to be $[\alpha]_D -10.5^{\circ}$, a value considerably higher than that ($[\alpha]_D -5.9^{\circ}$) of the sodium salt of the corresponding propyl derivative (Kipping, Trans., 1908, 93, 465). The oily, optically impure salt of the *d*-acid was treated in a similar manner, and the solution of the resulting sodium salt examined polarimetrically, but only qualitatively; it was dextrorotatory, and showed a deviation of about $+0.5^{\circ}$.

The resolution of the original *dl*-sulphonic acid was also accomplished in a similar manner by fractionally crystallising its salt with *d*-methylhydrindamine. In this way, we obtained pure *d*-methylhydrindamine *l*-sulphobenzylethylisobutylsilicyl oxide (m. p. $207-209^{\circ}$), the salt of the *l*-acid remaining in an optically impure state as an oil in the aqueous-alcoholic mother liquors. From the pure *dAdB*-methylhydrindamine salt we prepared the sodium salt of the *d*-acid, which, in aqueous solution, had a specific rotation of $[\alpha]_D +10.4^{\circ}$.

The methylhydrindamine salts of the optically active sulphobenzylethylisobutylsilicyl oxides are, on the whole, very similar to the corresponding derivatives of the active sulphobenzylethylpropylsilicyl oxides, and yet they differ from the latter in one or two respects. The *dAdB*- and *lAlB*-salts are well-defined, crystalline compounds, but the *dAlB* and the *lAdB*-salts do not crystallise from aqueous solvents, and have only been obtained as oils. A mixture of the *dAdB*- and *AlB*-salts melts at about 203° , and does not liquefy at a much lower temperature when it is suddenly heated, as does a mixture of the corresponding sulphobenzylethylpropylsilicyl compounds.

¹ The melting points and specific rotations of some salts of the *dl*-acid and of the two optically active compounds are given in the following table:

Salts of dl-, d-, and l-Sulphobenzylethylisobutylsilicyl Oxides,
 $\text{SO}_3\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{SiEt}(\text{C}_4\text{H}_9)_2\cdot\text{O}\cdot\text{SiEt}(\text{C}_4\text{H}_9)_2\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}.$

d-Methylhydrindamine Salts.

<i>d</i> -Acid.	<i>l</i> -Acid.
M. p. 207—209°.	An oil.
$[\alpha]_D^{20} + 16.7^\circ$ in methyl alcohol.	
$[\alpha]_D^{20} + 14.8^\circ$ in chloroform.	

l-Methylhydrindamine Salts.

<i>d</i> -Acid.	<i>l</i> -Acid.
An oil.	M. p. 207—209°.
	$[\alpha]_D^{20} - 17.0^\circ$ in methyl alcohol.
	$[\alpha]_D^{20} - 14.4^\circ$ in chloroform.

l-Menthylamine Salts.

<i>d</i> -Acid.	<i>dl</i> -Acid.	<i>l</i> -Acid.
M. p. 254—258°	M. p. 240—245°	M. p. 253—257°
$[\alpha]_D^{20} - 13.9^\circ$ *	$[\alpha]_D^{20} - 14.7^\circ$	$[\alpha]_D^{20} - 17.2^\circ$

d-Bornylamine Salts.

<i>d</i> -Acid.	<i>dl</i> -Acid.	<i>l</i> -Acid.
M. p. 209—211°	M. p. 207—209°	M. p. 210—212°

Cinchonidine Salts.

<i>d</i> -Acid.	<i>dl</i> -Acid.	<i>l</i> -Acid.
M. p. 171—173°	M. p. 175—177°	M. p. 181—183°
$[\alpha]_D^{20} - 68.5^\circ$	$[\alpha]_D^{20} - 69.3^\circ$	$[\alpha]_D^{20} - 69.3^\circ$

Cinchonidine Hydrogen Salts.

<i>d</i> -Acid.	<i>dl</i> -Acid.	<i>l</i> -Acid.
M. p. 282—284°	M. p. 225—229°	M. p. 229—231°

It will be seen from these data that the *l*-menthylamine salts of the *d*- and *l*-acids differ appreciably in specific rotation, although they have practically the same melting point; also, that the salt of the *dl*-acid melts at a considerably lower temperature than those of the active acids. In these respects the properties of the *l*-menthylamine salts of the *d*-, *l*-, and *dl*-sulphobenzylethylisobutylsilicyl oxides afford a much clearer indication of the enantiomorphous relationship of the active acids than do the corresponding salts of the sulphobenzylethylpropylsilicyl oxides, which are practically identical in melting point and specific rotation (*loc. cit.*, p. 468).

* This and all the following values for specific rotations are those obtained in methyl-alcoholic solution.

The *d*-bornylamine salts of the *d*-, *l*-, and *dl*-acids have practically the same melting point, and are very similar in outward properties, so that in these compounds the enantiomorphous character of the acids is almost completely hidden, as is the case with the corresponding sulphobenzylethylpropyl derivatives.

The cinchonidine salts of the *d*- and *l*-acids show a greater difference in melting point than do the corresponding salts of the sulphobenzylethylpropylsilicyl oxides, and it may be noted that the salt of the *d*-acid has a lower melting point than that of the *dl*-compound. The cinchonidine hydrogen salts are very similar in properties, and show the same relationship as that observed in the case of the sulphobenzylethylpropyl compounds.

The broad results of this investigation bring out, as was to be expected, the analogy between the derivatives of benzylethylisobutylsilicil and those of its lower homologue. As regards the general chemical behaviour of the two series of compounds, the most important difference between them is observed in sulphonating the silicil and the oxide, in which process the *isobutyl* derivatives are doubtless decomposed to some extent with separation of the *isobutyl* group (*loc. cit.*, p. 2014). As regards the optical properties of the active acids, it is obvious that the substitution of *isobutyl* for *propyl* in the two asymmetric silicon groups nearly doubles the molecular rotation, and also has an effect in emphasising the enantiomorphous character of the compounds when they are combined with a given optically active base.

EXPERIMENTAL.

Resolution of dl-Sulphobenzylethylisobutylsilicyl Oxide.

When a solution of *l*-methylhydrindamine hydrochloride is added slowly to an aqueous solution of the pure sodium salt of sulphobenzylethylisobutylsilicyl oxide, prepared from the pure *l*-menthylamine salt, there is first formed a precipitate resembling the white of an egg; on stirring the liquid, however, this precipitate disappears, and the solution becomes exceedingly viscous. On further addition of the hydrochloride, the solution again becomes turbid, and finally there is formed an oily precipitate, which becomes solid on standing for some hours. After decanting the supernatant liquid and washing with water, the precipitate is dissolved in aqueous methyl alcohol and the solution allowed to remain at the ordinary temperature; a white, crystalline deposit impregnated with oil is thus obtained. This deposit is separated by filtration, freed from oil as far as possible with the aid of a filter-pump, and recrystallised from aqueous methyl alcohol as before. Continued fractional crystallisation results in the separation of the original precipitate into colourless crystals of the pure *l*AIB-

salt, intermediate fractions, and mother liquors which give oily deposits of the optically impure *dAdB*-salt. The intermediate crystalline fractions are reserved, and worked up with corresponding portions of salt obtained in subsequent resolutions. The oily deposits from the mother liquors are mixed with a very slight excess of sodium carbonate, and the methylhydrindamine is expelled with the aid of steam, alcohol being added from time to time to control the very troublesome frothing which always occurs. The solution of the sodium salt of the optically impure *d*-acid is then neutralised with acetic acid, concentrated on the water-bath, and treated with a solution of *d*-methylhydrindamine hydrochloride; the precipitate, after having been washed with water, is fractionally crystallised from aqueous methyl alcohol, and is thus resolved into pure *dAdB*-salt, intermediate crystalline deposits, and mother liquors containing the *lAdB*-compound. Employing the *l*- and *d*-bases alternately in this way, a very considerable proportion of the *dl*-acid may be resolved and obtained in the form of the pure *lAdB*- and *dAdB*-salts. The most convenient method is to convert half the original *dl*-acid into the salt of the *d*-base, and the other half into the salt of the *l*-base; after isolating as large a quantity as possible of the *dAdB*- and *lAdB*-compounds, the bases in the remaining salts are transposed in the manner described above, and fractional crystallisation is continued.

d-Methylhydrindamine *d*-Sulphobenzylethylisobutylsilicyl Oxide,
 $\text{SO}_3\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{O}\cdot\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}\cdot 2\text{C}_{10}\text{H}_{18}\text{N}$.

This compound is deposited from aqueous-alcoholic solution in opaque nodules; it is very soluble in alcohol and in moist acetone, but almost insoluble in anhydrous acetone or water. It melts at $207-209^\circ$, and is anhydrous.

Its specific rotation was determined in methyl-alcoholic solution:

0.756, made up to 25 c.c. in a 2-dcm. tube, gave $\alpha + 1.01^\circ$; whence
 $[\alpha]_D + 16.7^\circ$.

Its specific rotation in chloroform solution was also determined:

0.756, made up to 25 c.c. in a 2-dcm. tube, gave $\alpha + 0.89^\circ$; whence
 $[\alpha]_D + 14.8^\circ$.

l-Methylhydrindamine *l*-Sulphobenzylethylisobutylsilicyl Oxide.

This compound is, of course, identical with the *dAdB*-salt in melting point and outward properties. Its specific rotation was determined in methyl-alcoholic solution:

0.695, made up to 25 c.c. in a 2-dcm. tube, gave $\alpha - 0.95^\circ$; whence
 $[\alpha]_D - 17.0^\circ$.

Its specific rotation was also determined in chloroform solution :

0.695, made up to 25 c.c. in a 2-dcm. tube, gave $\alpha - 0.8^\circ$; whence $[\alpha]_D - 14.4^\circ$.

A mixture of approximately equal quantities of the *dAdB*- and *lAlB*-methylhydrindamine salts melted at about 203° ; on plunging the mixture into a bath heated at 160° , no sign of change occurred, and the melting point was practically the same as before. This behaviour is different from that of a mixture of the corresponding salts of sulphobenzylethylpropylsiliclyl oxide, as in the case of the latter a mixture of the two pure salts has a melting point considerably lower than that of its components, and liquefies immediately when introduced into a bath already heated at 160° . In all other respects, however, these salts of the active sulphobenzylethylisobutylsiliclyl oxides are very similar to those of their lower homologues.

Sodium Salts of the Optically Active Sulphobenzylethylisobutylsiliclyl Oxides.

The sodium salts of the *d*- and the *l*-acids are crystalline compounds very readily soluble in water. For the determination of their specific rotations, weighed quantities of the pure *d*- and *l*-methylhydrindamine salts were separately dissolved in alcohol, treated with a slight excess of hydrated sodium carbonate, and the organic base distilled as rapidly as possible in a current of steam. When all the base had been removed, the solutions were transferred to an evaporating basin, neutralised with acetic acid, and concentrated to about 10 c.c. They were then filtered if necessary, made up to 20 c.c., and examined :

0.923 of the *dAdB*-methylhydrindamine salt (= 0.663 gram of sodium salt) gave, in a 2-dcm. tube, $\alpha + 0.69^\circ$; whence $[\alpha]_D + 10.4^\circ$.

0.823 gram of the *lAlB*-methylhydrindamine salt (= 0.591 gram of sodium salt) gave, in a 2-dcm. tube, $\alpha - 0.62^\circ$; whence $[\alpha]_D - 10.5^\circ$.

The agreement between these values leaves nothing to be desired, and corresponds with a molecular rotation, $[M]_D \pm 65.9^\circ$, which is very much higher than that, $[M]_D \pm 35.2^\circ$, of the sulphobenzylethylpropylsiliclyl oxides.

In preparing these sodium salts, care was taken to avoid heating the alkaline solutions longer than was absolutely necessary, in view of the possibility of the occurrence of racemisation; the following experiment seems to show, however, that the acids are optically stable.

A solution of the sodium salt, obtained as described above from the *dAdB*-methylhydrindamine salt, was treated with a solution of *d*-methylhydrindamine hydrochloride; the precipitate solidified almost

immediately, and after one crystallisation from aqueous methyl alcohol melted at $206-208^{\circ}$, that is to say, at practically the same temperature as the pure *d*AB-compound.

l-Methylhydrindamine *d*-Sulphobenzylethylisobutylsilicyl Oxide.

This compound is precipitated as an oil on adding a solution of *l*-methylhydrindamine hydrochloride to a solution of the sodium salt of the *d*-acid; as it did not solidify even when kept for some weeks, it was only examined qualitatively. It is soluble in cold water, but is precipitated from the solution on the addition of sodium chloride. Methyl alcohol also produces a turbidity in the aqueous solution, but the liquid becomes clear again on the further addition of the alcohol. The effect of heat is similar; the solution becomes turbid at first, but clear again on heating more strongly.

d-Methylhydrindamine *l*-Sulphobenzylethylisobutylsilicyl Oxide.

This compound, prepared from the solution of the sodium salt of the *l*-acid, showed the same behaviour as its *d*AB-isomeride.

The great difference in outward properties between the salts obtained by combining one of the optically active sulphobenzylethylisobutylsilicyl oxides with *d*-methylhydrindamine, on the one hand, and with the *l*-base on the other, is even more marked than in the case of the corresponding active sulphobenzylethylpropylsilicyl oxides, and the fact that a given solution of one of the active acids yields such very different compounds when treated under similar conditions with *d*- and with *l*-methylhydrindamine hydrochloride respectively, is in itself sufficient proof of the enantiomorphous structure of the silicon molecule.

l-Menthylamine *d*-Sulphobenzylethylisobutylsilicyl Oxide,
 $\text{SO}_3\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{O}\cdot\text{SiEt}(\text{C}_4\text{H}_9)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}\cdot 2\text{C}_{10}\text{H}_{21}\text{N}.$

This salt is obtained as a crystalline precipitate on adding a solution of *l*-menthylamine hydrochloride to a solution of the sodium salt of the *d*-acid; it crystallises from moist ethyl acetate in clusters of shining needles, which contain water of crystallisation:

0.8096 lost 0.0626 at 100° . $\text{H}_2\text{O} = 7.7$.

$\text{C}_{46}\text{H}_{84}\text{O}_7\text{N}_2\text{S}_2\text{Si}_2\cdot 4\text{H}_2\text{O}$ requires $\text{H}_2\text{O} = 7.4$ per cent.

The anhydrous salt melts at $254-258^{\circ}$.

The specific rotation was determined in methyl-alcoholic solution: 0.747 of the anhydrous salt, made up to 20 c.c., gave $\alpha = 1.04^{\circ}$ in a

2 -dm. tube; whence $[\alpha]_D = 13.9^{\circ}$.

l-Menthylamine 1-Sulphobenzylethylisobutylsilicyl Oxide.

This compound, prepared from the sodium salt of the *l*-acid, is similar in outward properties to the corresponding salt of the *d*-acid : 0.5974, of air-dried salt, lost 0.045 at 100°. $H_2O = 7.5$ per cent.

The anhydrous compound melts at 253—257°.

The specific rotation of the salt was determined in methyl-alcoholic solution in a 2-dcm. tube :

0.552 of anhydrous salt, made up to 20 c.c., gave $\alpha = -0.95^\circ$; whence $[\alpha]_D = -17.2^\circ$.

Although these two menthylamine salts have practically the same melting point, they have appreciably different specific rotations; their melting points are also considerably higher than that of the salt of the *dl*-acid. A mixture of equal quantities of the salts of the *d*- and *l*-acids was crystallised from ethyl acetate; the deposit thus obtained melted at the same temperature as the original salt of the *dl*-acid. No indication of dimorphism was observed in the case of these menthylamine salts, which is rather noteworthy considering their very close relationship to the corresponding derivatives of the sulphobenzylethylpropylsilicyl oxides (Kipping, Trans., 1908, 93, 468).

d-Bornylamine Salts of the d- and l-Acids.

These salts were precipitated on the addition of *d*-bornylamine hydrochloride to solutions of the sodium salts of the active acids in the form of oils which ultimately solidified. They were washed with water and recrystallised from aqueous methyl alcohol, being thus obtained in beautiful, shining needles. The salt of the *d*-acid melted at 209—211°, and that of the *l*-acid at 210—212°; the melting point of the salt of the *dl*-acid (this vol., p. 2015) is a trifle lower than that of the *d*-compound.*

Cinchonidine Salts of the d- and l-Acids.

These salts were obtained as sticky precipitates on the addition of cinchonidine hydrochloride to solutions of the sodium salts of the active acids; the compounds were washed with water, dried, and then crystallised over sulphuric acid from acetone containing a little methyl alcohol; they were thus obtained as colourless, crystalline powders.

* This opportunity may be utilised to correct an obvious slip which was made in describing the *d*-bornylamine salts of the sulphobenzylethylpropylsilicyl oxides (Kipping, Trans., 1908, 93, 470). In the sentence (11 lines from the bottom of the page) "recrystallisation failed to raise the melting point of the *l*-acid as high as that of the salt of the *d*-acid," the letters *l*- and *d*- should be transposed.

Their specific rotations were determined in methyl-alcoholic solution with samples which had been dried at 100°.

Salt of *d*-acid: 0.314, made up to 25 c.c., gave $\alpha - 1.72^\circ$ in a 2-dm. tube; whence $[\alpha]_D - 68.5^\circ$.

Salt of *l*-acid: 0.357, made up to 25 c.c., gave $\alpha - 1.98^\circ$ in a 2-dm. tube; whence $[\alpha]_D - 69.3^\circ$.

The salt of the *d*-acid melted at 171—173°, and that of the *l*-acid at 181—183°, slight darkening occurring in both cases. In outward appearance and in their behaviour towards solvents, they resembled the salt of the *dl*-acid (*loc. cit.*, p. 2015).

Cinchonidine Hydrogen Salts of the d- and l-Acids.

These compounds were obtained from the normal cinchonidine salts in the manner described in the case of the *dl*-derivative (p. 2016). The friable solids thus obtained were dissolved in a small quantity of methyl alcohol, and precipitated by means of acetone in the form of microcrystalline powders. The salt of the *d*-acid melted at 232—234°, and that of the *l*-acid at 229—231°.

We desire to express our thanks to the Royal Society for a Government Grant in aid of this investigation.

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CCIX.—Some Molecular Compounds of Styphnic and Picric Acids.

By CHARLES STANLEY GIBSON.

ALTHOUGH our knowledge of the molecular compounds formed by picric acid with other aromatic substances is fairly extensive, little work has so far been done on similar compounds which may be formed by the analogous trinitroresorcinol, styphnic acid. Noelting and Salis (*Ber.*, 1882, 15, 1863) prepared naphthalene styphnate, and Summerer has recently described (*Diss.*, Zürich, 1907) cinnamaldehyde styphnate.

The styphnic acid used was successfully prepared according to Merz and Zetter's method (*Ber.*, 1879, 12, 2037), and the following compounds have been prepared.

Acenaphthene Styphnate, $C_{12}H_{10}, C_6H(OH)_2(NO_2)_3$.

When 3.1 grams of acenaphthene and 5 grams of styphnic acid are each dissolved in alcohol and the solutions mixed while still warm,

acenaphthene styphnate crystallises out in long needles on cooling. It is obtained pure by recrystallisation from alcohol:

0.1510 gave 14.05 c.c. N_2 at 16.7° and 754.5 mm.: $N = 10.88$.

$C_{18}H_{13}O_8N_3$ requires $N = 10.53$ per cent.

Acenaphthene styphnate crystallises in long, orange-coloured, doubly-refracting prisms, which melt at $153-154^\circ$ to a red liquid. It is moderately soluble in cold alcohol, and is decomposed by a large excess of this solvent; after several crystallisations, pure acenaphthene (m. p. $= 95^\circ$) is obtained. In acetone and ethyl acetate it is readily soluble and stable, but in ether it is partly decomposed. The compound is decomposed by carbon disulphide, chloroform, carbon tetrachloride, light petroleum or benzene.

Phenanthrene Styphnate, $C_{14}H_{10}C_6H(OH)_2(NO_2)_3$.

This compound is prepared by dissolving 3.6 grams of phenanthrene in alcohol, and adding the solution to 5 grams of styphnic acid, also dissolved in alcohol. On cooling, the substance separates out, and is easily purified by recrystallisation from alcohol:

0.1806 gave 15.10 c.c. N_2 at 18.5° and 763.3 mm. $N = 9.83$.

$C_{20}H_{13}O_8N_3$ requires $N = 9.93$ per cent.

Phenanthrene styphnate crystallises in yellowish-red, doubly-refracting needles melting at $125-126^\circ$. It is readily soluble and stable in alcohol, acetone or ethyl acetate. In benzene it is readily soluble, but the substance suffers decomposition. It is quickly decomposed by carbon disulphide, ether, chloroform or carbon tetrachloride in the cold, but more slowly by light petroleum.

α -Bromonaphthalene Styphnate, $C_{10}H_7BrC_6H(OH)_2(NO_2)_3$.

On adding 4.2 grams of α -bromonaphthalene to a warm alcoholic solution of 5 grams of styphnic acid, bromonaphthalene styphnate is obtained. The compound is recrystallised from as little alcohol as possible; the pure substance possesses a distinct odour of bromonaphthalene:

0.1704 gave 13.6 c.c. N_2 at 20.0° and 757.5 mm. $N = 9.27$.

$C_{16}H_{10}O_8N_3Br$ requires $N = 9.30$ per cent.

α -Bromonaphthalene styphnate melts at $107-108^\circ$, and crystallises in small, bright yellow, doubly-refracting needles. It is soluble in ether, acetone or ethyl acetate, and is stable in these solvents. When light petroleum is added to the ethereal solution, styphnic acid is precipitated. The compound is soluble and fairly stable in alcohol, being decomposed by a large excess of the solvent. Bromonaphthalene styphnate is immediately decomposed by carbon

2100 MOLECULAR COMPOUNDS OF STYPHNIC AND PICRIC ACIDS

disulphide, chloroform, or carbon tetrachloride, styphnic acid being precipitated in all cases. In benzene the decomposition is rapid.

m-Nitroaniline Styphnate, $\text{NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{NO}_2\cdot\text{C}_6\text{H}_3(\text{OH})_2(\text{NO}_2)_3$.

This substance is formed when warm alcoholic solutions of equimolecular proportions of *m*-nitroaniline and styphnic acid are mixed and the solution allowed to evaporate spontaneously. The compound is very soluble in alcohol, and is best recrystallised from hot benzene:

0.1242 gave 19.6 c.c. N_2 at 16.0° and 757.0 mm. $\text{N} = 18.56$.

$\text{C}_{12}\text{H}_9\text{O}_{10}\text{N}_5$ requires $\text{N} = 18.28$ per cent.

m-Nitroaniline styphnate crystallises in pale yellow, doubly-refracting needles, which, like the corresponding picrate, tend to form in radiate masses. It melts at 137° , and is soluble and stable in alcohol, ethyl acetate or benzene. From its solutions in ether or acetone the compound does not crystallise well. It is decomposed by carbon disulphide, and to some extent by chloroform or carbon tetrachloride.

m-Nitroaniline Picrate, $\text{NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{NO}_2\cdot\text{C}_6\text{H}_3(\text{OH})(\text{NO}_2)_3$.

This compound has not been previously described. It is prepared in exactly the same way as *m*-nitroaniline styphnate, and may be purified by recrystallising from alcohol or benzene:

0.0728 gave 12.2 c.c. N_2 at 21.0° and 761.2 mm. $\text{N} = 19.48$.

$\text{C}_{12}\text{H}_9\text{O}_9\text{N}_5$ requires $\text{N} = 19.08$ per cent.

m-Nitroaniline picrate crystallises in doubly-refracting, radiating needles, which melt at 143° . The crystals are of a distinctly deeper yellow colour than those of the styphnate, but the properties generally of the two substances show a very close resemblance. The crystals deposited from a benzene solution lose benzene of crystallisation very rapidly and become opaque. The compound is also readily soluble and stable in alcohol, ethyl acetate, ether or acetone, but it does not crystallise well from the latter solvent. *m*-Nitroaniline picrate is almost insoluble, and not so stable in carbon disulphide or carbon tetrachloride; in chloroform it is rather more soluble. Even in hot water the compound suffers very little decomposition. It is interesting to note in this connexion that Vignon and Évieux, basing their evidence on the heat of solution in benzene of *o*-nitroaniline and of picric acid compared with that of a preparation made by mixing picric acid with fused *o*-nitroaniline, and also on a cryoscopic determination of the molecular

weight of the preparation, have quite recently (*Compt. rend.*, 1908, 147, 69) concluded that no combination between picric acid and *o*-nitroaniline takes place.

It is hoped to continue this investigation.

I wish to express my thanks to Professor W. J. Pope, M.A., F.R.S., who suggested this work and supplied me with the materials employed.

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CCX.—*The Formation of Some Carbides.*

By JOHN NORMAN PRING.

ALTHOUGH a certain number of compounds between metals and the element carbon has been known from quite early times, the composition was, in almost all cases, ill-defined, and the chemistry of these substances was first systematically investigated by Moissan, who, at the high temperatures made available with the arc furnace, succeeded in producing a whole series of new compounds.

More recently the preparation of some of these carbides has been brought about by "thermite" reactions, and in ordinary fuel-heated furnaces.

In view of the fact that the carbide-forming elements are, in many cases, known to react with carbon monoxide and other carbon compounds, considerable discussion has recently been evoked as to whether these elements can combine directly to form carbides in the absence of such gaseous impurities.

Moissan showed that at very high temperatures, where carbon appeared to occur in the state of vapour, direct union with the vapour of some elements, such as silicon, was possible, and that at lower temperatures the presence of metals, such as iron in a fused condition, facilitates the formation of carbides in the case of boron and silicon. Moissan concludes that this is due to the metal acting as solvent of these elements (*The Electric Furnace*, London, 1904, pp. 264, 274).

The nature of the reaction which takes place at lower temperatures, namely, below 1200°, where carbon vapour can no longer be supposed to exist, has, however, been left an open question, and an estimation of the temperatures at which carbon first combines directly with other elements has not yet apparently been carried out.

A clearing up of these points has, it is hoped, been accomplished in the case of aluminium, silicon, and iron in the following work, which also includes some preliminary experiments with magnesium.

The method used consisted in mixing intimately the purified element and very carefully purified carbon, obtained in a fine state of division, and heating at a constant temperature for specified periods of time under diminished pressure, which, in this work, was in some cases as low as 0.01 mm.

The apparatus used was of two types: (1) For experiments below 900°, the mixture was placed in a porcelain boat, which was heated in a silica tube in a platinum-foil electric resistance furnace, a thermojunction being arranged alongside the tube and opposite the boat for the temperature readings. The ends of the silica tube were connected by ground joints to glass tubes, on one side leading to a Töpler pump, and on the other to a tube containing charcoal, which could be cooled by liquid air. In this way, the substance could be heated for several hours at a temperature constant to within about 10°, and as high as 900°, while the pressure remained below 0.03 mm. (2) For experiments above 900°, the mixture was placed in a graphite tube about 9 cm. long, 0.9 cm. external, and 0.6 cm. internal diameter, fitted in graphite end plugs, which were mounted in water-cooled brass holders in a tubular glass flask, the brass tubes being fitted into the side-tubes of the flask by means of wax. The graphite tube was heated by the passage of an electric current through it (compare Greenwood, *Trans.*, 1908, 93, 1485). Exact temperature readings could be taken by means of the Wanner optical pyrometer. A constant temperature, which could be taken as high as 2000°, was obtained in the middle part of the tube over a distance of about two-thirds of the total length, although the temperature at the ends was rather lower. The pressure could be kept at about 0.05 mm. by means of a glass tube 7 inches long and 1½ inches diameter, filled with coconut charcoal, and immersed in liquid air. This tube was usually attached to the neck of the flask by means of a ground joint. Several experiments were conducted without the use of the charcoal tube, exhausting by the Töpler pump alone, whereby the pressure obtained was not so low. Measurement of the low pressures was made by means of a McLeod gauge, which was first carefully calibrated.

The carbon used for studying the formation of carbides was in the form of retort graphite, which was powdered in an agate mortar, sieved through cotton, and heated in a carbon tube resistance furnace for about half an hour* at a temperature of 1800–2000° in a current

* Originally designed by Hutton and Patterson (*Trans. Faraday Soc.*, 1905, 1, 1).

of chlorine and then for the same time in hydrogen. Before this treatment, the carbon contained 0.38, and afterwards less than 0.01, per cent. of ash.

Aluminium Carbide.

Gantz and Masson (*Compt. rend.*, 1897, 134, 187) showed that aluminium in presence of a trace of aluminium chloride reacts with carbon monoxide and carbon dioxide to give aluminium carbide and alumina.

Franck (*Chem. Zeit.*, 1898, 22, 236) prepared a small quantity of this carbide by heating aluminium in carbon monoxide, and also by heating the metal with lampblack.

In the preparation of aluminium carbide in the Moissan arc furnace, starting with ingots of the metal, the author found that the reaction took place chiefly by the fixation of the carbon monoxide present (Pring, *Trans.*, 1905, 87, 1531).

Weston and Ellis (*Trans. Faraday. Soc.*, 1908, 4, 60) prepared aluminium carbide by the interaction of aluminium powder and various varieties of carbon, heated in a muffle furnace, and obtained in some cases yields as high as 66 per cent. of the carbide.

They conclude that the air played an important part in the reactions, chiefly by causing oxidation of the aluminium powder and thereby raising the temperature of the mixture, and if air had been rigorously excluded they consider that very little action would have taken place.

In the work described below, aluminium was used in the form of a very fine powder, carefully washed with alcohol and ether to remove any carbonaceous matter, and dried at 150°. The metal, on analysis, was found to contain Al = 98.91, Fe = 0.90, and Si = 0.04 per cent. This was then intimately mixed with purified carbon in the proportion indicated by the formula Al_4C_3 , and heated in the porcelain boat according to method 1, or in the graphite tube according to method 2.

After each experiment a portion of the product was examined by completely decomposing with dilute hydrochloric acid, collecting the gas evolved, and analysing in a Sodeau apparatus, which enabled the methane to be estimated to within about 0.05 per cent.

Precautions were taken to avoid the formation of nitric acid during explosion of the hydrogen.

The results of the experiments are expressed in tabular form on p. 2104.

From these results, one can see that the reaction commences at a surprisingly low temperature, namely, 650°. It proceeds, however, somewhat slowly, and only at about 1400° is the combination complete

Temp.	Duration of experiment.	Pressure.		Percentage of Al_4C_3 * in product.
		Maximum. mm.	Average. mm.	
1420°	75 mins.	9.0	6.0	nearly all Al_4C_3
1250	80 "	7.0	5.5	39.2
1250	80 "	0.05	0.04	48.4
1180	30 "	15.0	9.0	40.7
1090	30 "	6.0	5.0	20.7
970	30 "	8.0	7.0	9.4
870	50 "	1.0	0.8	4.4
750	2 hours	0.4	0.14	6.4
650	2 "	0.35	0.20	1.3
500	2 "	0.03	0.008	nil
600	20 "	0.12	0.015	nil

* Only some two-thirds of the mixture was exposed to the temperature here recorded in the case of experiments carried out above 900° where the graphite tube was used.

in a short time. The product in all cases is the characteristic, brilliant yellow-coloured Al_4C_3 , although, probably owing to its fine state of division, no crystalline structure was apparent when examined under a high power microscope.

Silicon Carbide.

The formation of this compound is stated by Moissan to take place when carbon and silicon are heated together in a wind furnace at a temperature of 1200—1400° (*The Electric Furnace*, 1904, p. 264).

Tucker (*J. Amer. Chem. Soc.*, 1906, **28**, 853) estimated the temperature of formation of silicon carbide in the electric carborundum furnace to be 1700—1800°.

Greenwood (*Trans.*, 1908, **93**, 1492) found that the reduction of silica by carbon takes place at 1460° under a pressure of 2—3 mm., the product obtained being carborundum.

Further investigation of the formation of this carbide was undertaken by the author in order to ascertain the precise temperature of the direct union and to examine the possible effect of the presence of solid and gaseous impurities. Various kinds of silicon were taken for this purpose, namely, that obtained by the Deville process with aluminium, commercial silicon prepared in the electric furnace, and thirdly, the same substance carefully purified by subsequent chemical treatment. This purification process consisted in grinding the silicon in an agate mortar and sieving through cotton, to obtain a very fine powder. This was then treated with hot aqua regia until no further action took place, filtered and washed, and then treated twice with hot hydrofluoric acid solution in a platinum dish and evaporated to dryness.

Analyses of the different varieties gave the following results :

	Deville silicon.	Commercial silicon.	Purified silicon.
Si	—	—	99.70
Fe	0.73	5.52	0.10
Al	5.24	0.73	0.20
SiO ₂ and other impurities	2.62	—	—

All the experiments with silicon were conducted in the electrically heated graphite tube inside the glass flask, and the product was afterwards analysed by taking a known weight, treating with hot aqueous potassium hydroxide to remove the free silicon, and then heating at a red heat in air until no further loss in weight occurred, to estimate the carbon. With impure silicon, a further treatment with aqua regia followed, to remove iron, etc.

The product remaining consisted of the carbide, SiC, which had a crystalline appearance when examined under a microscope, and was of a light grey colour when the purified silicon was used and greenish-black with the impure varieties. The results are given below in tabular form.

In experiment No. 9, electrolytic iron to the amount of 13 per cent. of the total mixture was added to the silicon and carbon to ascertain any effect in lowering the temperature of combination was reduced. The result showed that the presence of iron causes no difference in this reaction.

Expt. No.	Temp.	Duration of run, mins.	Nature of silicon.	Pressure.		Percentage of SiC in product.
				Maximum. mm.	Average. mm.	
1	1100°	12	Commercial	1.00	0.35	nil
2	1300	30	"	0.15	0.10	28.0
3	1200	40	"	0.14	0.10	5.5
4	1280	30	Deville	0.02	0.02	2.1
5	1400	30	"	0.02	0.015	30.0
6	1200	35	"	Atmosphere of CO		1.2
7	1310	30	Purified	0.05	0.03	8.0
8	1400	30	"	0.07	0.05	30.0
9	1240	30	Purified + 13% Fe	4.00	3.00	nil
10	1340	30	Purified	5.00	4.00	8.0

It is thus apparent that with highly purified silicon combination occurs at temperatures as low as 1300°, whilst already at 1400° a considerable proportion of the materials have united to form carborundum. With the more impure varieties, the reaction commences at somewhat higher temperatures.

Iron Carbide.

Moissan considered that the direct union of carbon, silicon, and boron with iron at 1200° indicates that these elements exert a slight vapour pressure at this temperature (*The Electric Furnace*, 1904, p. 258).

The union of carbon with iron in a vacuum has been investigated by W. C. Roberts-Austen (*J. Iron and Steel Inst.*, 1890, 81), who heated together electrolytic iron and diamond dust, and found that some combination took place at a full red heat. No exact temperature measurement was made, however, and no information regarding the completeness of the vacuum appears to be given. It was accordingly thought it would be of some interest to carry out further experiments on this subject at various known temperatures, using the highest vacuum obtainable in the apparatus described above, and measuring the pressure.

Electrolytic iron in the form of fine powder was mixed with carefully purified retort carbon (containing very little ash) in the proportion demanded by Fe_3C , placed in a porcelain boat, and heated according to method 1 described above.

After each experiment, a small portion of the product was tested for combined carbon by the colorimetric test, using 50 per cent. nitric acid, and comparing the colour with that given by a steel of known composition, treated alongside in the same manner.

Temp.	Duration of run.	Pressure.		Percentage of combined carbon in product.
		Maximum. mm.	Average. mm.	
870°	3 hours	0.05	0.04	0.44
850	3 "	0.06	0.05	0.32
660	5 "	0.08	0.05	nil
750	6 "	0.04	0.02	0.35

Magnesium.

The existence of a carbide of magnesium was first announced by Berthelot, who heated magnesium powder in a stream of acetylene and obtained a product which evolved acetylene on treatment with water. This carbide was found by Moissan to be completely decomposed at a high temperature.

By the reduction of magnesium oxide by carbon, Slade (*Trans.* 1908, 93, 327) showed the possibility of preparing metallic magnesium by the use of a graphite retort. On conducting this reaction in a current of hydrogen, the metallic product was largely contaminated with a carbide which gave acetylene on treating with water. Slade considers this carbide to have arisen by the interaction of acetylene and magnesium.

Weston and Ellis (*Trans. Faraday Soc.*, 1908, 4, 71) publish footnote to a paper announcing that evidence of the direct combination of magnesium and carbon has been obtained.

In the present work, it was thought that it might be possible to effect decomposition of magnesium chloride vapour at a high

temperature by means of hydrogen containing some acetylene or benzene vapour by an analogous reaction to that which was found to take place between silicon chloride and acetylene, giving silicon carbide.

Some experiments were accordingly carried out* in a carbon tube furnace placed horizontally, fitted in the centre with a vertical inlet tube, and provided with a condensing tube at the end. The carbon tube was heated to the temperature at which magnesium chloride volatilises, a current of hydrogen containing benzene vapour passed through, and magnesium chloride admitted by the vertical tube. It was, however, not found possible to effect any decomposition or obtain a product which gave any considerable evolution of gas on treating with acid.

The effect of heating pure magnesium powder with pure carbon in a high vacuum was next investigated, using method 1, described above. It was found in all cases that the metal practically all distilled from the boat, leaving behind carbon, and condensing in the cooler parts of the porcelain tube. The product was analysed by decomposing with hydrochloric acid, collecting the gas evolved, and estimating the hydrocarbon present. No trace of acetylene gas in any case noticeable. On treating with hot water, a slight evolution of gas was noticed with the product from experiment 4.

Small amounts of a hydrocarbon, other than acetylene, appeared to be present in this gas, apparently showing a slight solubility of carbon in magnesium at these temperatures, with the formation of traces of some carbide other than that obtained at higher temperatures.

Expt. No.	Temp.	Duration of run.	Pressure.		Percentage of (Mg_2C) ? in product.
			Maximum. mm.	Average. mm.	
1	650°	30 mins.	0·8	0·25	0·85
2	700	2 hours	0·2	0·10	0·6
3	1000	45 mins.	0·20	0·15	0·4
4	600	3 hours	0·05	0·04	1·2

Summary and Conclusions.

Silicon.—The direct union of pure silicon and carbon in a vacuum commences between 1250° and 1300°, the reaction proceeding rapidly above 1400°.

Commercial silicon containing 5 per cent. of iron and 0·7 per cent. aluminium reacts with carbon at all temperatures above 1200°.

The presence of iron does not apparently facilitate the reaction, nor does carbon monoxide exert any influence between the limits of

* Conducted by W. Fielding.

atmospheric pressure and 0.03 mm.; hence already below its melting point silicon is proved to combine with carbon.

Aluminium.—This element unites directly with pure carbon in a vacuum at its melting point (650°), forming aluminium carbide (Al_4C_3), and the velocity of the reaction increases with the temperature, proceeding rapidly above 1400° .

Iron.—Direct union with carbon occurs at about 700° under a pressure of about 0.05 mm.

Magnesium.—Evidence is forthcoming to indicate the formation to a limited extent of a new carbide below 600° , this compound giving some saturated hydrocarbon (probably methane) on decomposition with water or acids. At higher temperatures the reaction apparently ceases. This unstable compound is probably Mg_2C , of an analogous nature to Al_4C_3 ; the only carbide of magnesium, MgC_2 , hitherto known corresponds with the carbides of the alkali and alkaline earth metals.

Finally, I wish to thank Dr. R. S. Hutton for the kind interest he has taken in this work and for much valuable advice and assistance.

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CCXI.—*The Relation between Absorption Spectra and Chemical Constitution. Part XII. Some Amino-aldehydes and -ketones of the Aromatic Series.*

By EDWARD CHARLES CYRIL BALY and EFFIE GWENDOLINE MARSDEN.

In a previous paper (Baly and Collie, *Trans.*, 1905, **87**, 1332), the absorption spectra of benzaldehyde and acetophenone were described, and it was shown that both these compounds exhibit only a very small absorption band, the view being put forward that the motions of the benzene ring were to a very great extent restrained by the presence of the ketonic oxygen in the β -position in the side-chain. We have now investigated some of the amino-aldehydes and -ketones of the benzene series with the view of determining the influence of the amino-group. From a comparison of the absorption of aniline with that of benzene, it is evident that the influence of the amino-group is directly opposite to that of the ketonic oxygen in the β -position, for the very large absorption band of aniline would suggest that the dynamic activity of this compound is even greater than that of

benzene itself. This fact is, of course, in agreement with the results obtained by magnetic rotation (Perkin, *Trans.*, 1896, 79, 1025). In the amino-aldehydes and -ketones we have therefore two well-defined influences brought simultaneously to bear on the phenyl group, namely, first the ketonic oxygen in the β -position tending to restrain and lock up the motions of the phenyl group, and secondly, the amino-group with an influence of exactly the opposite nature.

Inasmuch as the absorption curves of these compounds all show large absorption bands, it is evident that the restraining influence of the ketonic or aldehydic group is more than counteracted by the amino-group.

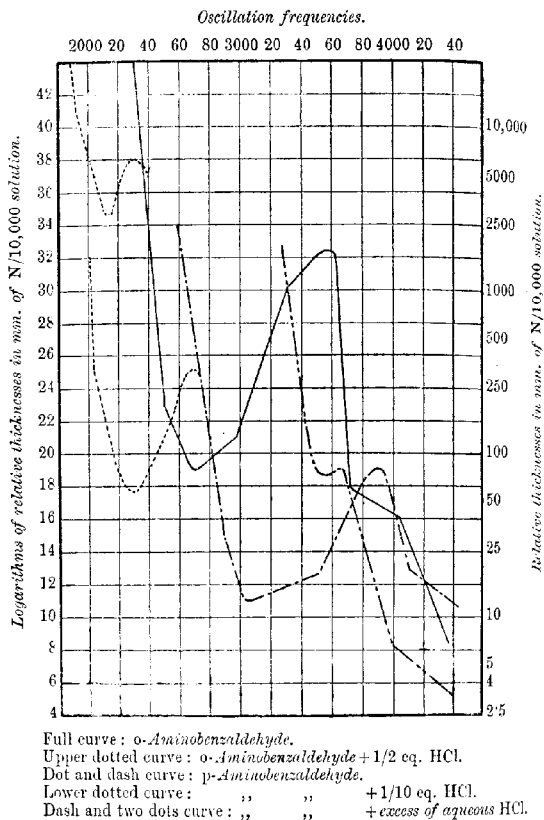
It is interesting, moreover, to note that in these compounds generally the ortho-isomeride exhibits the greatest absorption band and the para the least; the meaning of this is not at present clear. It may be pointed out that these substances are coloured yellow, owing to their absorption bands being situated in the visible region of the spectrum. If to the solution of one of the aminoaldehydes or aminoketones in alcohol a small quantity of alcoholic hydrogen chloride is added, the yellow colour of the solution is very much intensified, and the absorption spectrum now shows a new absorption band nearer to the red and at greater concentration than the original band. In the presence therefore of very small quantities of hydrogen chloride, the alcoholic solution of amino-aldehydes and -ketones shows two absorption bands, the principal band being unaltered by the small amount of free mineral acid.

The effect of the mineral acid is most pronounced in the para-isomeride and least in the case of the meta-compound. In Fig. 1 the full curve shows the absorption spectrum of *o*-aminobenzaldehyde, and the dot and dash curve that of the para-isomeride; the upper and lower dotted curves show the absorption of the two compounds in presence of 1/2 equiv. and 1/10 equiv. of hydrogen chloride respectively. The greater effect produced in the case of the para-compound by 1/10 equiv. of hydrogen chloride than by as much as 1/2 equiv. in the case of the ortho-isomeride is very evident. The lower portions of both curves are not altered in any way by such small quantities of acid. Fig. 2 (full and dotted curves) and Fig. 3 (dot and dash, dash and two dots curves) represent the absorption of *p*- and *o*-aminoacetophenone in neutral and slightly acid alcoholic solutions respectively. We give only the results obtained with the ortho- and para-compounds; the results were quite similar with the meta-isomerides, but in a less degree than in the case of the ortho-derivative.

The amount of acid necessary depends very much on the nature of the compound, and it appears that less acid is required with the aldehydes than with the ketones, and, moreover, as has already

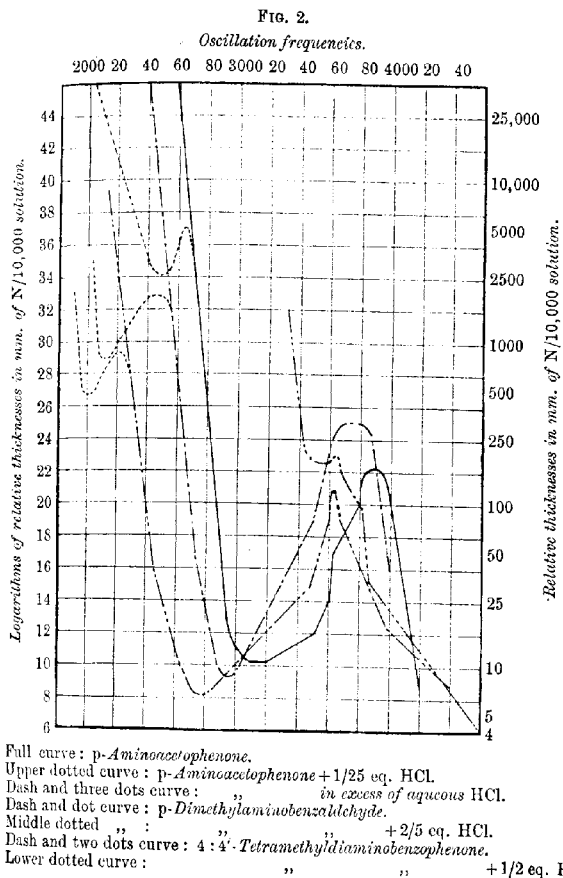
been pointed out, less is required with the para-compound than with the two other isomerides. The most sensitive compound is *p*-amino-benzaldehyde, which develops a large and very persistent absorption band with only 1/10 equiv. of hydrogen chloride, and, since the band

FIG. 1.



is obtained with $N/1000$ solution, so the concentration of the acid is only $N/10,000$. The extreme sensitiveness of the reaction is thus manifest. If too much hydrogen chloride is added, the effect is destroyed, owing to the conversion of the compounds into the hydrochlorides, which in every case are perfectly white compounds. The

maximum amount allowable is of the order of $1/2$ equiv. ; more than this quantity causes the whole spectrum to tend towards that of the hydrochloride. Indeed, in the curve of *o*-aminobenzaldehyde with $1/2$ equiv. of hydrogen chloride (Fig. 1, upper dotted curve), it will be noticed



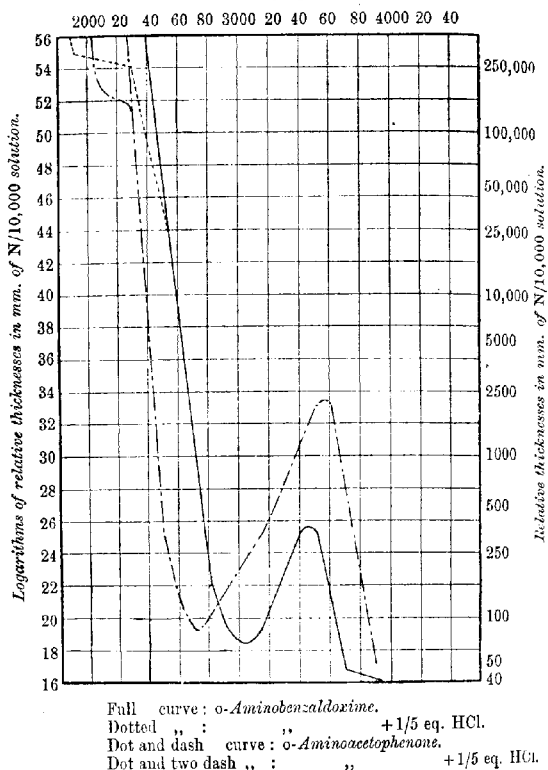
that it does not quite coincide with the curve of the compound in neutral solution; this is owing to the partial conversion into the hydrochloride.

If the aminic hydrogen atoms be replaced by alkyl groups, the same

results are obtained, although in a less degree. This can be seen from the curves of *p*-dimethylaminobenzaldehyde (Fig. 2, dot and dash and middle dotted curves) and of 4:4'-tetramethyldiaminobenzophenone (Fig. 2, dash and two dots and lower dotted curves).

If the substances are dissolved in water or dilute alcohol, the above

FIG. 3.
Oscillation frequencies.



effect is no longer produced on the addition of hydrochloric acid; the spectrum merely tends towards that of the hydrochloride, although this latter spectrum is only finally reached in presence of considerable excess of acid. The absorption of *p*-aminobenzaldehyde hydrochloride and of *p*-aminoacetophenone hydrochloride is shown in Fig. 1

(dash and two dots curve) and Fig. 2 (dash and three dots curve) respectively.

The effect of the addition of hydrogen chloride to these compounds seems to be due undoubtedly to the amino-group. Some experiments at present in progress on certain complex amino-compounds would support this view, for it appears that the nature of the substituent group in the aniline residue is immaterial. For example, the amino-benzaldoximes exhibit the same phenomenon as can be seen from the full and dotted curves on Fig. 3, which show the absorption of *o*-aminobenzaldoxime. It would appear that the addition of the acid produces an increase in the amount of the residual affinity of the amino-group; in neutral solution the nitrogen atom is undoubtedly trivalent, and possibly the addition of the acid makes it functionate as a quinquivalent atom, so that its residual affinity is increased in the ratio of 5 to 3. This increase in the residual affinity, being as it were superadded to that of the neutral molecule, would no doubt account for the new absorption bands. The increase in the residual affinity produced by the addition of acid would account also for the catalytic action of mineral acid on the acetylation of amino-compounds.

Conclusions.

1. The amino-aldehydes and -ketones of the aromatic series in neutral alcoholic solution exhibit one broad absorption band.
2. The addition of small quantities of alcoholic hydrogen chloride to the solution causes the development of a second absorption band, the first remaining unaltered, provided that insufficient acid has been added to produce the hydrochloride.
3. This effect is most marked in the amino-aldehydes and further in the para-isomeride. It is not produced at all in aqueous solution.
4. The phenomenon appears to be due to the amino-group; it is possible that the nitrogen atom is caused to functionate as a quinquivalent atom, so that the residual affinity is increased in the ratio of 5 to 3.
5. The results throw considerable light on the catalytic action of mineral acid in the acetylation of amines.

The authors wish to express their thanks to the Chemical Society for a grant in aid of this work.

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CCXII.—*The Affinity of Certain Alkaloids for Hydrochloric Acid.*

By VICTOR HERBERT VELEY.

It has been observed for many years that certain alkaloids, such as those of the cinchona and *nux vomica* groups and others, although containing two atomic proportions of nitrogen or two amino-residues, yet combine for the most part with only one molecular proportion of hydrogen chloride, their dihydrochlorides being obtained either with difficulty under special conditions, or even under no conditions, in the solid form. It was thought that the methyl-orange method could be applied to determine the proportion of hydrochloric acid which will combine in solution with the monohydrochlorides of such alkaloids, and hence some insight might be obtained into the relative affinities of the two nitrogen atoms or amino-groups. I have at the outset to express my obligations to Mr. David Howard for beautiful samples of certain cinchona hydrochlorides, to Messrs. Burroughs, Wellcome and Co., and workers in their laboratories, for other samples, and to several friends who have also rendered assistance.

The Cinchona Group.

It is well known that the salt formed directly from these alkaloids and hydrochloric acid is the monohydrochloride, B, HCl , sometimes called the normal salt, and that the hydrochloride, $B, 2HCl$, sometimes called the acid salt, can be obtained by evaporation, under special conditions, of solutions of the alkaloid or mono-salt with excess of concentrated hydrochloric acid, or by passing the dried gas over the mono-salt or other indirect methods.

Hesse (*Annalen*, 1875, 176, 227) and Oudemans (*ibid.*, 1876, 182, 51) determined the specific rotatory powers, $[\alpha]_D$, of aqueous solutions of these alkaloids with different molecular proportions of hydrochloric acid, but drew no conclusions as to any difference between the affinity values of the two amino-groups. Colson and Darzens (*Compt. rend.*, 1893, 118, 250) remarked that quinine contains a feeble basic function analogous to that of quinoline, and a stronger basic function similar to that of the amines of the ethylic or piperidine series. Salamonsen (*Ned. Tydsch. Pharm.*, 1895, 7, 195, 225) regarded quinine as a true dibasic alkaloid, and the combination of one molecule of alkaloid with two molecules of hydrogen chloride as the normal salt, on the ground that if the salt were truly of an acid character it would not be neutral to methyl-orange, even although it is acid to litmus. More precise information from the physical

chemistry standpoint is afforded by the determinations of Berthelot and Gaudechon (*Compt. rend.*, 1903, **136**, 128) of the thermo-neutrality values of the cinchona alkaloids, which may be summarised as follows (ϕ = heat quantity in 100 gram calories).

Quinine and Quinidine.

$B + 2 \text{ mols. HCl.}$	$\phi = 208$
$B + 1 \text{st mol. HCl.}$	$\phi = 119$

$$(\text{Difference}) B + 2 \text{nd mol. HCl.} \quad \phi = 89$$

Cinchonine and Cinchonidine.

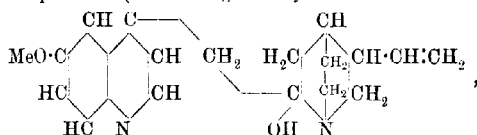
$B + 2 \text{ mols. HCl.}$	$\phi = 202.0$
$B + 1 \text{st mol. HCl.}$	$\phi = 131.5$

$$(\text{Difference}) B + 2 \text{nd mol. HCl.} \quad \phi = 70.5$$

No values could be obtained for cinchonamine, owing to the sparing solubility of its salts.

The heats of neutralisation of the former pair of bases with the first molecular proportion of hydrogen chloride are approximately equal to that of dimethylamine ($\phi = 118$), and those of the second pair of bases are approximately equal to those of methylamine ($\phi = 131$) or piperidine ($\phi = 135$), whereas in the case of the second molecular proportion of hydrogen chloride the values are rather higher than that of quinoline ($\phi = 68$). The difference in the affinity values of the two amino-groups is very marked.

If the formula of Königs (*Annalen*, 1906, **347**, 232) be adopted for quinine and quinidine (cinchinine), namely :



and that of cinchonine and cinchonidine which differs from the above only in containing a hydrogen atom in the place of the methoxyl group, then all these four bases contain a piperidine and a quinoline residue, and consequently the affinity constant of one amino-group should be of the order of piperidine, $k_b = 1.58 \cdot 10^{-3}$, and the other of the order of quiniline, $k_b = 3.4 \cdot 10^{-3}$.

It was first found that the monohydrochlorides of the five alkaloids, quinine, quinidine, cinchonine, cinchonidine, and cinchonamine, showed no indications of hydrolysis with $N/20$ or $N/40$ solutions ($V = 4 \times 10^3$ and 8×10^3), and thus they resemble piperidine hydrochloride in that their affinity constants are beyond the limit which can be detected by

the methyl-orange method, which is approximately of the order $k_b = 1 \times 10^{-7}$, but as these alkaloids are precipitated by ammonia from their salts, their affinity value is less than 1×10^{-5} .

Each of the hydrochlorides was then dissolved in such a volume of $N/20$ hydrochloric acid so as to form an $N/20$ or $N/40$ solution (according to solubility) of the dihydrochloride on the presumption that no hydrolysis of the latter salt took place. Such solutions were diluted after the lapse of two or three days in accordance with my usual plan of working, and hence the amount of such hydrolysis and the affinity constants of the second amino-group determined. The results are given as under:

Quinine Dihydrochloride.—Two solutions were examined at a temperature of 15° :

$V = 4 \times 10^4$.	$V = 2 \times 10^4$.	$V = 4 \times 10^4$.	$V = 2 \times 10^4$.
1.0	2.0	4.9	8.2
1.9	4.0	6.9	8.2
3.1	6.0	7.9	8.2
4.0	8.2		

Values of $k = 1$ and 2, hydrolysis value = 31.2 , $k_{b(15)} = 3.32 \cdot 10^{-10}$.

Quinidine Dihydrochloride.—Two solutions were examined at slightly different temperatures.

$V = 4 \times 10^4$. Temp. 11° .	$V = 2 \times 10^4$. Temp. 13° .	$V = 4 \times 10^4$. Temp. 11° .	$V = 2 \times 10^4$. Temp. 13° .
0.9	1.9	3.4	7.6
1.8	3.8	4.5	9.5
2.6	5.7	5.3	11.4

Values of $k = 0.9$ and 1.9 , hydrolysis values = 29.7 and 28.1 respectively, $k_{b(11)} = 3.17 \cdot 10^{-10}$, and $k_{b(13)} = 3.19 \cdot 10^{-10}$.

It appears that quinidine is a slightly weaker base than quinine, but the difference is almost within the limit of experimental error.

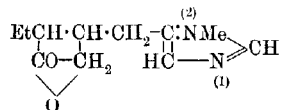
The results obtained with the dihydrochlorides of cinchonine, cinchonidine, and cinchonamine were practically identical with those obtained with the salt of quinine; hence it is not necessary to give the figures; the only difference was that all reaction sooner came to an end in the case of cinchonamine.

The conclusion to be drawn from these experiments is that the affinity constant of the second amino-group is rather less than that of quinoline (see following paper), although it was to be expected that the constant would be rather greater, as the piperidine nucleus is in the 4-position with respect to the nitrogen atom. But otherwise the results are in general accordance with those of Colson and Darzens and of Berthelot and Gaudechon, but the statement of Salamonsen,

that an aqueous solution of quinine dihydrochloride is neutral to methyl-orange, appears to require modification; it is probable that the last author used a too concentrated solution of the indicator. So far as I am aware, no determinations have been made of the electric conductivity of the dihydrochlorides of these alkaloids which might serve as a further confirmation of the above results; Bredig (*Zeitsch. physikal. Chem.*, 1894, **13**, 289) determined the conductivities of quinine, quinidine, and cinchonine monohydrochlorides, from which it was concluded that the velocity of the cinchona ion is of a relatively low order. It may be added that Hädrich (*Zeitsch. physikal. Chem.*, 1893, **12**, 476), by determinations of the specific rotatory power of certain salts of the cinchona group, arrived at the conclusion that ionisation is complete at $N/10$ or $N/20$ dilution. If, then, these observations, as also mine, that no hydrolysis could be detected at the latter dilution, are well founded, then it would follow either that Robertson's view (*J. Physical Chem.*, 1907, **11**, 437) is correct, that accurate hydrolysis values can only be obtained when the respective functions of the base and hydrochloric acid are of a sufficient different order, or, what may come to the same thing, the velocity of the cinchona ion is so small that its detection by the methyl-orange method requires infinite time.

Pilocarpine, $C_{10}H_{16}O_3N_2$.

The investigations of Jowett, published in a series of memoirs (*Trans.*, 1900—1905), have shown that this alkaloid forms a stable monohydrochloride, $B.HCl$, from which the base can be displaced by a solution of ammonia in excess (*Trans.*, 1900, **77**, 477), and also that the alkaloid and its isomeride, *isopilocarpine*, react with methyl iodide to form a methiodide, from which methylamine can be obtained, but no further methylation takes place (*Trans.*, 1906, **89**, 497, 854) even when heated with excess of methyl iodide. The constitution of the alkaloid is probably represented by the formula:



(*Trans.*, 1905, **87**, 797; compare Pinner and Schwarz, *Ber.*, 1902, **35**, 2441), or, in other words, it is a methylglyoxaline with a lactonic, namely, the homopilopic, residue attached. From these considerations it would appear that pilocarpine contains one amino-group of affinity value lower than that of ammonia, $k_b = 1.7 \cdot 10^{-5}$, probably the residue marked $\cdot\text{N}^{\cdot}$, whilst that of the other group, $\cdot\text{NMe}$, would

(1)

(2)

probably be of a much lower order as associated with an acidic group, and hence might take up hydrogen chloride only under special conditions.

I am indebted to Dr. Jowett for a sample of pilocarpine (mono) hydrochloride used to obtain the following results:

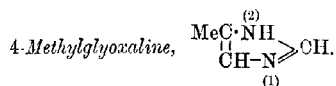
First, it was found that the above salt showed no hydrolysis in $N/20$ solution by the methyl-orange method; hence the affinity value of the one amino-group is greater than $k_b = 1 \cdot 10^{-7}$, and would be intermediate between this number and $1 \cdot 7 \cdot 10^{-5}$ (see above).

Secondly, the monohydrochloride was dissolved in such a volume of $N/20$ hydrochloric acid so as to obtain an $N/20$ solution of the dihydrochloride, presuming no hydrolysis took place, and otherwise the method of procedure was similar to that used for the cinchona salts.

The following results were obtained at 16° :

$V = 8 \times 10^4$	$V = 4 \times 10^4$	$V = 8 \times 10^4$	$V = 4 \times 10^4$
1.0	2.1	4.1	8.3
2.1	4.2	5.2	8.3
3.2	6.3	6.3	8.3

Hence $k = 1.05$ and 2.1 respectively, hydrolysis value $= 65.6$ and $k_{b(10)} = 4.24 \cdot 10^{-11}$, a value not far removed from that of creatinine, $k_{b(40)} = 3.69 \cdot 10^{-11}$; the two amino-groups in pilocarpine have therefore a very widely different affinity value, being in the ratio $1 : 500,000$ as a rough approximation, and the results from the physical chemistry standpoint confirm the conclusions arrived at from chemical considerations only:



Having regard to the relationship of pilocarpine to the glyoxaline series, it was thought that a study of the affinity values of the nitrogen atoms or amino-groups of a glyoxaline might be a matter of additional interest. For this purpose, Dr. Jowett kindly supplied me with a sample of the above compound. The material presented slight experimental difficulties, in that it appeared to retain traces of water or an oil, and as it was volatile at a comparatively low temperature it could not be dried by any heating process, but by adopting suitable precautions, it was believed that the necessary small quantities were weighed out with only a slight error.

First, it was found that when an $N/10$ solution of hydrochloric acid was added to an aqueous solution of a weighed quantity of the base, the point of neutrality corresponded very approximately with the formation of the monohydrochloride when methyl-orange was used as

an indicator according to the usual process of volumetric analysis, when the mass of reacting salt is very greatly in excess of that of the methyl-orange.

Secondly, it was found that when the base was dissolved in the volume of $N/20$ hydrochloric acid required to form an $N/20$ solution of the monohydrochloride, B, HCl , such a solution showed no trace of hydrolysis by the methyl-orange method; hence the affinity value of the grouping $\cdot N \cdot$ is greater than $1 \cdot 10^{-7}$, and is probably of the order of the aliphatic amines.

Thirdly, the base was dissolved in a volume of $N/20$ hydrochloric acid so as to form an $N/20$ solution of the dihydrochloride, $B, 2HCl$, presuming that no hydrolysis took place; the solution was very strongly acidic towards methyl-orange, which would show that the second grouping, $\cdot NH \cdot$, was either acidic or that the dihydrochloride was very largely hydrolysed, such as the cases of hydrazine or the diamines of the aromatic amines, for example. After standing for some hours, the solution was diluted to $N/103$ — $N/400$, and these solutions allowed to stand for about eighteen hours.

The following results were obtained at a temperature of 18° :

I. $V=8 \times 10^4$.	II. $V=4 \times 10^4$.	III. $V=2 \times 10^4$.	I. $V=8 \times 10^4$.	II. $V=4 \times 10^4$.	III. $V=2 \times 10^4$.
0.6	1.1	2.45	2.3	7.0	11.3
1.2	2.3	4.9	2.3	8.3	11.3
1.3	4.7	7.35	2.3	9.2	11.3
2.3	5.9	9.8			

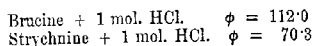
Values of $k=0.6$, 1.2 , and 2.45 , corrected 0.605 , 1.21 , and 2.42 respectively; hydrolysis value 37.8 ; $k_{(18)}=2.78 \cdot 10^{-10}$. As the solutions gave results identical within the limits of experimental error with those obtained in the case of hydrazine hydrochloride, hydrolysis value = 37.5 (Trans., 1908, 93, 660), the $N/20$ original solution as heated for three hours at 60° and diluted, as previously described, the results obtained did not differ from those of the solution which had not been so treated. The general conclusion appears to be that as above methylglyoxaline behaves as a diamine; unfortunately, there have been no determinations of the heats of neutralisation of this base with one and two molecular proportions of hydrogen chloride respectively, but it seems probable that the heat value for the first molecule of hydrogen chloride would be about 100, and that for the second molecule would be less than 1. It would obviously have been better for the purpose of comparison with pilocarpine to have examined a glyoxaline of the type

$$\begin{array}{c} \text{RC:NMe} \\ | \\ \text{HC}-\text{N} \gg \text{CH} \end{array}$$

The Nux Vomica Alkaloids: Strychnine and Brucine.

Both the above alkaloids form a crystalline hydrochloride, B_2HCl , although containing two atomic proportions of nitrogen or two amino-groups. So far as investigations have gone, it appears that they contain (i) a hydropyridine residue, (ii) a grouping convertible into quinoline (Hanssen, *Ber.*, 1886, **20**, 451; Stocker, *ibid.*, 810), and (iii) a grouping $-O\text{<} \begin{smallmatrix} C(OH)^- \\ N^- \end{smallmatrix}$, transformable by an intramolecular

rearrangement into $-O\text{<} \begin{smallmatrix} CO^- \\ NH \end{smallmatrix}$, in that the so-called methyl- and benzyl-strychnines, for example, may be regarded as betaine derivatives of strychnic acid (Tafel, *Annalen*, 1891, **264**, 33; Monfang and Tafel, *ibid.*, 1898, **309**, 49). The thermochemical investigations of Berthelot and Gaudechon (*Compt. rend.*, 1905, **140**, 715) show that strychnine and brucine may be regarded as monobasic, although the latter will under certain conditions take up as many as four molecular proportions of hydrochloric acid; their neutrality values given are as follows:



It is therefore evident that brucine is the stronger.

It was found that the hydrochlorides of brucine and strychnine showed no hydrolysis by the methyl-orange method; hence the affinity value of one amino-group in both is greater than 1×10^{-7} ; as it has been shown independently that ammonia in solution will displace strychnine but not brucine from its salts, the affinity value of one amino-group in brucine is greater, and in strychnine less, than $1.7 \cdot 10^{-5}$. The method of determining the affinity value of the second amino-group in these alkaloids was precisely as described above, and the following results were obtained.

Strychnine.—Two solutions of the monohydrochloride, dissolved in the required quantity of $N/20$ hydrochloric acid to form a dihydrochloride, were examined, and the following results obtained at 20° :

$V = 8 \times 10^4$.	$V = 4 \times 10^4$.	$V = 8 \times 10^4$.	$V = 4 \times 10^4$.
1.0	2.2	5.3	10.9
2.1	4.2	6.2	13.0
3.1	6.4	7.4	13.0
4.2	8.6	8.6	13.0

Values of $k = 1.05$ and 2.15 , corrected 1.06 and 2.13 respectively. hydrolysis value = 66.3 , hence $k_{(20)} = 5.95 \cdot 10^{-11}$, a value of the order of benzobetaine, $3.4 \cdot 10^{-11}$ (Cumming, *Proc. Roy. Soc.*, 1906, **78**, A. 139); this result appears to confirm the views of Tafel.

Brucine.—The method of experiment was precisely as that used for strychnine, and the following results obtained at 20° :

$V = 8 \times 10^4$	$V = 4 \times 10^4$
1.3	2.6
2.5	5.1
3.7	7.8

but it was observed that all reaction sooner came to an end.

Values of $k = 1.25$ and 2.6 , corrected 1.27 and 2.55 ; hydrolysis value = 79.4 , hence $k_{h(20)} = 2.52 \cdot 10^{-11}$. The affinity value of the second amino-group in brucine is approximately half the corresponding value of strychnine; thus the position of affairs is reversed as compared with the first amino-group, a result which has been independently confirmed by another method. However, the data obtained by the methyl-orange method confirm the general conclusions which have been arrived at regarding the constitution of these alkaloids.

Gelsemine.

The formula assigned by Cushny (*Ber.*, 1893, 26, 1713) to the hydrochloride of this alkaloid is $C_{49}H_{83}O_{11}N_5 \cdot 2HCl$; although more extended investigation may possibly lead to its revision, yet it is not probable that the ratio of the atomic proportion of nitrogen to molecular proportion of hydrogen chloride would be reduced to equality, and therefore the alkaloid might come under the same category as those discussed above. An $N/40$ solution of the above salt showed no trace of hydrolysis by the methyl-orange method; such a volume of $N/20$ hydrochloric acid was then added to a given volume of the solution so as to obtain an $N/40$ solution of a possible salt, B_4HCl . But on dilution and subsequent examination in the usual way, it was found that none of the hydrochloric acid thus added had combined with the alkaloid; thus proving that of the five atomic proportions of nitrogen, three possess a different function from the remaining two, presuming that Cushny's formula is substantially correct.

Summary.

(1) Determinations are given of the affinity values of the nitrogen atoms or amino-groups of certain alkaloids which, although containing more than one atomic proportion of nitrogen, yet form stable combinations with one molecular proportion of hydrogen chloride only, the dihydrochloride being produced under special conditions, or, not as yet, obtained in the solid form. It is shown by the application of the methyl-orange method that in all cases these values are of a widely different order.

(2) In the case of the alkaloids of the cinchona group, the affinity

value of one amino-group is less than that of a hydropyridine, whilst that of the other is rather lower than that of quinoline. These results are in conformity with the accepted constitution of these alkaloids and the thermo-neutrality values for the first and second molecular proportion of hydrogen chloride added.

(3) In the case of pilocarpine, the value of one group is intermediate between that of ammonia and the limit value of the methyl-orange method, whilst that of the other is equal to those of true amphoteric electrolytes. A methylglyoxaline was examined on account of the relationship of pilocarpine to these compounds; the value of the two nitrogen atoms or amino-groups and its general behaviour corresponded with a diamino-base of the type of hydrazine.

(4) Of the *Nux vomica* alkaloids, strychnine and brucine were examined; in both cases the affinity value of one amino-group was beyond the limit value of the methyl-orange method, but it has been shown independently that the value of the brucine group is greater than that of strychnine. The affinity value of the second amino-group is of the order of value of the betaines, which accords with the conclusions arrived at from chemical considerations; but, here, the state of affairs appears to be reversed, as the grouping in brucine has a slightly lower value than that of strychnine.

I have again to express my obligation to friends, and it is hoped to lay before the Society in the near future a method for further differentiating between the basic values of the alkaloids.

CCXIII.—*The Affinity Constants of Bases as Determined by the Aid of Methyl-Orange.*

By VICTOR HERBERT VELEY.

Introduction.

IN the present communication it is proposed to give an account of results obtained by the methyl-orange method for the hydrochlorides of nitrogenous bases derived from cyclic compounds. As some of them were antecedent to those contained in my previous publication (this vol., p. 652, *et seq.*), but deferred for the sake of convenience, there is nothing further to add as to variation of method or improvement in detail.

Since my investigations were published, Salm (*Zeitsch. physikal.*

Chem., 1908, 63, 83, *et seq.*) has used various indicators, and among them methyl-orange, for determining the affinity constants of acids; the process adopted by this chemist differs from mine in detail rather than in principle, in that he used the method commonly known as nesslerisation, namely a series of standard tints. My method appears to possess the advantage that several determinations and not one only can be made with the same solution, and thus the chance of error reduced proportionally to the number of such observations, but, on the other hand, Salm's method would have the advantage over mine when the hydrochloric acid, either as such or liberated from a chloride by the process of hydrolysis, exceeds a certain amount, since experience has shown that a methyl-orange solution containing too many of the red ions (whatever their constitution) cannot be matched as regards tint by a length of solution containing the orange ions only. Or to state the case in another form, observations by my method fail when the absorption bands $\lambda=541$, between D and F , and $\lambda=504$, between b and F , characteristic of the reddened methyl-orange, become too pronounced.

The values obtained for the affinity constants of bases by my method are more generally in accordance with those obtained by the catalysis of methyl acetate or inversion of sucrose methods; this result is to be expected, as in all three methods the constants are determined inferentially by the amount of chemical change conditioned, whether instantly or after the lapse of time, by the hydrochloric acid liberated by hydrolysis. Objections have been raised to this inferential method of calculation by Robertson (*J. Physical Chem.*, 1907, 11, 437) on the ground that approximately accurate results can only be obtained if the larger function, namely, that of the hydrochloric acid, is sufficiently great as compared with the smaller function, that of the base. This criticism has been met by Lundén (*J. Biol. Chem.*, 1908, 4, 268) on mathematical considerations based on the degree of ionisation of each of the several constituents present in a solution of a hydrolysed hydrochloride. So far as my method is concerned, it would appear that there is a certain cogency in Robertson's criticism, in that in the case of bases with affinity constants of the order of $k_b = 1 \times 10^{-3}$ only a few observations could be obtained before a condition of equilibrium set in.

The values obtained by my method are also in fair accordance with those obtained by the electrolytic method, by which the affinity constants are determined directly. The values obtained by Farmer and Warth (*Trans.*, 1904, 85, 1713) by the extraction method in some cases accord with, but in others differ widely from, those furnished by other methods; herein it may be remarked that results obtained by any extraction method are of the nature of a double convergent series, on the

one hand, towards totality as regards the extracting liquid, and, on the other, towards *nil* as regards the extracted liquid.* The final state of affairs may be reached in some cases after some minutes, and in others after some hours, according to the peculiar circumstances of each case.

The experimental results are given in Section I, their mutual relationships discussed in Section II, and the temperature-coefficients of certain functions deduced from my experiments and those of others are dealt with in Section III.

Salts and Solutions used.

Some of the salts were purchased directly from reliable firms and purified by recrystallisation when necessary, and I have again to express my obligations to the Research Fund Committee of the Chemical Society for a grant towards the expense. Other samples were presented by various kind donors, alluded to in the sequel; others were prepared by adding concentrated hydrochloric acid solution to bases, either purchased or presented, and recrystallising the hydrochloride two or three times from water or alcohol. In cases in which the salts could not be conveniently obtained in a crystalline form by these methods, a weighed amount of the base was dissolved in an *N*/20 hydrochloric acid solution so as to form an *N*/20 solution of the hydrochloride, presuming that no hydrolysis occurred; such solutions were kept for a few days before further dilution, so that the final state of equilibrium of salt-base-acid-water, or ions thereof, might be reached.

EXPERIMENTAL.

Section I.—Benzonoid Amines: (1) Monoacidic bases.

Aniline Hydrochloride.—Solutions of three concentrations were examined, with the following results:

I. $V=4 \times 10^4$ Temp. 15°.	II. $V=2 \times 10^4$ Temp. 15°.	III. $V=2 \times 10^4$ Temp. 12°.	I. $V=4 \times 10^4$ Temp. 15°.	II. $V=2 \times 10^4$ Temp. 15°.	III. $V=2 \times 10^4$ Temp. 12°.
1.0	2.2	3.9	5.0	10.8	15.9
2.2	4.4	8.1	6.0	12.8	15.9
3.1	6.6	12.0	7.1	12.8	15.9
4.1	8.8	15.9	7.9	12.8	15.9

Values of k for I and II = 1 and 2.15, corrected 1.03 and 2.07;

* The use of chloroform, which has also been suggested as the extractive liquid in investigations of this kind, appears to be peculiarly open to criticism on account of the difficulty of maintaining this liquid free from traces of hydrochloric acid, so readily formed by exposure to light.

hydrolysis value = 32.2^* ; for III, $k = 4$; hydrolysis value = 31.2 ; $k_b = 2.6 \cdot 10^{-10}$ at 12° , $3.2 \cdot 10^{-10}$ at 15° .

As the hydrolysis values obtained at the several dilutions do not differ within the limits of experimental error, which may be taken as at \pm one unit, it would appear that at a dilution of $V = 1 \times 10^4$, or thereabouts, either the process of hydrolysis commences to be reversed, or the increased mass of inactive water may act as matter in the way of the smaller mass of water, which may be regarded as active, a conclusion which would be in accordance with the results obtained in the study of the effect of water on certain chemical changes. But with the view of meeting an obvious criticism that the methyl-orange method is not of a sufficient order of accuracy to differentiate between the amounts of hydrolysis at the several dilutions, the following calculations may be adduced.

In the Arrhenius equation

$$k_b/k_w = (1-x)V/x^2 \dots \dots \dots (1),$$

the value of the function k_b/k_w for aniline from the results of the most trustworthy observers is $(63 \pm 0.5)10^3$ at 15° ; substituting the values $V = (1, 2, 4)10^4$ respectively, and solving the equations for x , there are obtained numbers which multiplied by 100 are given in column II of the table below, and compared therewith the values given in column III, which are obtained by my method without applying corrections even of the first order. *

I.	II.	III.
V .	$100 x$ (calc.).	$100 x$ (found).
1×10^4	32.7 at 15°	32.2 at 12°
2×10^4	42.7 „	31.3 at 15
4×10^4	55.6 „	33.6 „

It will be evident that the differences of the last two values in column II are ten to twenty times greater than the differences of the uncorrected values in column III, and such result appears to be a sufficient answer to the criticism.

Derivatives of Aniline.

Monomethylaniline Hydrochloride.—Two concentrations were examined, with the following results (temperature 18°):

$V = 8 \times 10^4$.	$V = 4 \times 10^4$.	$V = 8 \times 10^4$.	$V = 4 \times 10^4$.
0.6	1.3	3.0	6.0
1.2	2.5	3.6	7.2
1.8	3.7	3.6	8.5
2.4	4.9	3.6	9.6

* As the methyl-orange solution used is the same as in my former work, the factors given on p. 656 were used; for each solution and individual observer, such factors must be determined separately. It appears desirable to call attention to this point, as my method is now being applied for the quantitative determination of acid solutions which are beyond the limit of volumetric analysis.

Values of $k=0.6$ and 1.2 ; hydrolysis value= 37.5 ; value of $k_b=2.55 \cdot 10^{-10}$.

It appears from this result that methylaniline is a weaker base than aniline; Walker (Trans., 1898, 67, 576) arrived at an opposite conclusion by the inversion of sucrose method at a temperature of 60° .

Dimethylaniline Hydrochloride.—The examination of this salt presented certain experimental difficulties, owing to the readiness with which the base separates out on exposure of the crystals to air; Walker (*loc. cit.*) was unable to obtain any results for this reason.

Two samples were examined at different times with different methods of manipulation, but the results obtained were the same within the limits of experimental error; two series of observations are given (temperature 18°):

$V=4 \times 10^4$.	$V=2 \times 10^4$.
1.2	2.2
2.4	4.5
3.6	6.6

Values of $k=1.2$ and 2.25 , corrected 1.16 and 2.3 ; hydrolysis value= 36.3 ; $k_b=2.42 \cdot 10^{-10}$. It will be observed that dimethylaniline is a slightly weaker base than aniline, although all reaction sooner comes to an end.

My results for the three bases given above are in the order of their thermoneutrality values given by Vignon (*Compt. rend.*, 1888, 106, 1668).

Monoethylaniline Hydrochloride.—As the salt of this base and the diethyl derivative cannot be conveniently obtained in a pure crystalline form, the bases were dissolved in hydrochloric acid to form an $N/20$ solution, which was subsequently diluted. Two dilutions were examined (temperature 19°):

$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=4 \times 10^4$.	$V=2 \times 10^4$.
0.9	1.75	3.6	5.25
1.9	3.5	4.4	5.25
2.7	5.25		

Values of $k=0.9$ and 1.75 ; hydrolysis value= 27.8 ; $k_b=4.17 \cdot 10^{-10}$.

Diethylaniline Hydrochloride.—No appreciable hydrolysis could be detected by my method, and therefore the constant is of the order $k_b=1 \times 10^{-7}$, or greater.

This result is in accordance with that obtained by Bredig (*Zeitsch. physikal. Chem.*, 1894, 13, 380) by the electric conductivity method; his values show that diethylaniline is a stronger base than any other benzenoid amine examined by him.

The Chloroanilines.—Some years ago Beilstein and Kurbatoff (Annalen, 1875, 176, 27) stated that the salts of the ortho-base were much more readily hydrolysed at a temperature of boiling water than those of the meta- and para-bases; the affinity constants of the meta- and para-bases have been determined by Farmer and Warth (*loc. cit.*).

o-Chloroaniline Hydrochloride.—Two dilutions were examined, but the colour reactions were not so sharp as in other cases; after the addition of three or four portions of the solution to the methyl-orange solution, some secondary change appeared to ensue (temperature 19°):

$V=8 \times 10^4$.	$V=4 \times 10^4$.
1.45	2.75
2.9	5.5
4.35	8.25

Values of $k=1.45$ and 2.75 , corrected 1.42 and 2.83 ; hydrolysis value $=90.3$; value of $k_b=9.16 \cdot 10^{-13}$.

m-Chloroaniline Hydrochloride.—Two dilutions were examined at slightly different temperatures:

Series I.	Series II.	Series I.	Series II.
$V=8 \times 10^4$.	$V=4 \times 10^4$.	$V=8 \times 10^4$.	$V=4 \times 10^4$.
Temp. 13°.	Temp. 10°.	Temp. 13°.	Temp. 10°.
1.5	2.7	5.7	10.7
2.0	5.4	7.1	13.2
4.3	7.9	8.5	15.6

Value of k in series I $=1.4$; in II $=8.3$; hydrolysis values 87.5 and 13 ; value of $k_{b(13)}=7.65 \cdot 10^{-12}$, of $k_{b(10)}=6.58 \cdot 10^{-12}$.

p-Chloroaniline Hydrochloride.—The results obtained at two dilutions are as follows (temperature 10°):

$V=8 \times 10^4$.	$V=4 \times 10^4$.	$V=8 \times 10^4$.	$V=4 \times 10^4$.
1.25	2.4	6.3	9.6
2.7	4.8	7.4	9.6
3.9	7.2	8.6	9.6
5.1	9.6	9.9	9.6

Values of $k=1.25$ and 2.4 , corrected 1.23 and 2.46 ; value of $k_b=1.24 \cdot 10^{-11}$; the last number is less than that, $12.7 \cdot 10^{-11}$ (corrected), given by Farmer and Warth (*loc. cit.*) at a temperature of 25° , which would correspond with a value, $7.6 \cdot 10^{-11}$, approximately, at a temperature of 10° .

Bromoanilines.—The salts of the meta- and para-bases were prepared as a crystalline form in the usual manner.

m-Bromoaniline Hydrochloride.—Two dilutions were examined, but

a difficulty similar to that noticed in the *o*-chloroaniline salt was experienced (temperature 19°):

$V=8 \times 10^4$.	$V=4 \times 10^4$.	$V=8 \times 10^4$.	$V=4 \times 10^4$.
0.9	1.7	3.7	6.9
1.8	3.5	4.7	6.9
2.7	5.2		

Values of $k=0.9$ and 1.75, corrected 0.88 and 1.77; hydrolysis value = 56.9; $k_{b(19)} = 9.5 \cdot 10^{-11}$.

p-Bromoaniline Hydrochloride.—The results obtained (temperature 18°) for two dilutions are as follows:

$V=8 \times 10^4$.	$V=4 \times 10^4$.	$V=8 \times 10^4$.	$V=4 \times 10^4$.
0.75	1.4	3.0	5.7
1.5	3.9	3.7	5.7
2.3	4.4		

Values of $k=0.75$ and 1.45, corrected 0.74 and 1.47; hydrolysis value = 46.1; $k_{b(18)} = 2.07 \cdot 10^{-10}$.

Benzylamine and the Toluidines.

Benzylamine Hydrochloride.—The hydrolysis of the salt was too small for accurate measurement; its behaviour, as to be expected, resembles that of the aliphatic amines, which have a thermoneutrality value equal to it. I find myself unable to follow the line of reasoning of Walker (*Proc. Roy. Soc.*, 1906, 88, 141) with regard to any peculiarity in the constant of the base.

o-Toluidine Hydrochloride.—The results obtained with this salt were given in my previous communication (pp. 657—658) as illustrative of the method; it is therefore unnecessary to repeat them.

m-Toluidine Hydrochloride.—Three series of experiments were conducted with solutions of this salt, the results of which are given below:

I.	II.	III.	I.	II.	III.
$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.	$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
Temp. 13°.	Temp. 13°.	Temp. 14°.	Temp. 13°.	Temp. 13°.	Temp. 14°.
0.9	1.8	3.6	4.6	7.0	14.2
1.9	3.6	7.2	5.4	7.0	14.2
2.8	5.4	10.7	6.2	7.0	14.2
3.6	7.0	14.2			

Values of $k=0.9$, 1.8, 3.6 respectively; hydrolysis value = 28.1; $k_{b(13)} = 3.54 \cdot 10^{-10}$, $k_{b(14)} = 3.9 \cdot 10^{-10}$. These last values are in accordance with that given by Bredig (*loc. cit.*), namely, $k_{b(25)} = 6.1$; that obtained by Farmer and Warth, $k_{b(23)} = 8.49 \cdot 10^{-11}$, is probably too low.

p-Toluidine Hydrochloride.—Two concentrations were examined at

temperature = 15° , but all reaction came sooner to an end than in the case of the isomeric salts :

$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=4 \times 10^4$.	$V=2 \times 10^4$.
1.0	1.8	3.8	3.6
2.1	3.6	4.6	3.6
2.9	3.6		

Values of $k=0.9$ and 1.8 respectively; hydrolysis value = 28.1 ; $k_{h(15)}=4.5 \cdot 10^{-10}$; this last number is more in accordance with that given by Bredig (*loc. cit.*), $k_{h(25)}=14 \cdot 10^{-10}$, than that, $k_{h(25)}=3.1 \cdot 10^{-9}$, obtained by Denison and Steele (Trans., 1906, 89, 999, 1386) by their ion migration method.

Substituted Toluidines.—The dimethyl-*o*- and *p*-toluidine hydrochlorides, like the ethylanilines, cannot conveniently be obtained in a crystalline condition; consequently, a similar method of procedure was adopted.

Dimethyl-o-toluidine Hydrochloride.—Two solutions were examined (temperature 15°):

$V=1 \times 10^4$.	$V=2 \times 10^4$.
0.9	1.9
1.9	3.9
2.7	5.7

Values of $k=0.95$ and 1.9 respectively; hydrolysis value = 11.9 ; $k_{h(15)}=3.08 \cdot 10^{-9}$.

Dimethyl-p-toluidine Hydrochloride.—Condition the same as above :

$V=2 \times 10^4$.	$V=1 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
0.7	1.3	3.6	3.9
1.4	2.6	4.2	3.9
2.2	3.9	4.8	3.9
2.9	3.9		

Values of $k=0.7$ and 1.3 , corrected 0.68 and 1.35 ; hydrolysis value = 8.4 , $k_{h(15)}=6.36 \cdot 10^{-9}$.

The behaviour of the above substances resemble the substituted anilines in that all reaction soon comes to an end; their affinity constants have not been previously determined.

Xylidines.

The hydrochlorides of *m*-4- and *p*-xylidine were prepared from their respective bases and examined.

m-4-Xylidine Hydrochloride.—Two solutions gave the following results (temperature 15°):

$V=2 \times 10^4$.	$V=1 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
1.4	2.8	4.2	8.4
2.8	5.6	5.4	8.4

Values of $k=1.4$ and 2.8 respectively; hydrolysis value $=21.9$,
 $k_{b(15)}=6.34 \cdot 10^{-10}$.

p-Xylidine Hydrochloride.—Two solutions were examined at 20° :

$V=2 \times 10^4$	$V=1 \times 10^4$	$V=2 \times 10^4$	$V=1 \times 10^4$
1.5	3.1	6.5	9.4
3.2	6.4	8.6	9.4
4.9	9.4		

Values of $k=1.6$ and 3.15 respectively; hydrolysis value $=24.8$,
 $k_{b(20)}=9.63 \cdot 10^{-10}$.

ψ -Cumidine Hydrochloride.—Two solutions were used (temperature 18°); all reaction soon came to an end:

$V=2 \times 10^4$	$V=1 \times 10^4$
0.7	1.6
1.4	2.8
1.4	4.2

Values of $k=0.7$ and 1.4 respectively; hydrolysis value $=10.9$,
 $k_{b(18)}=4.8 \cdot 10^{-9}$; this last is rather higher than that found, $k_{b(20)}=1.7 \cdot 10^{-9}$, by Lowenherz (*Zeitsch. physikal. Chem.*, 1898, 25, 385) by the solubility method.

Phenylhydrazine Hydrochloride.—Examination of solutions of this salt presented certain difficulties, owing to the separation of the base or its hydrate, a fact previously noted by other observers as characteristic of substituted hydrazines. Two concentrations were examined, but all reaction soon came to an end (temperature 15°):

$V=2 \times 10^4$	$V=1 \times 10^4$
1.0	1.8
1.9	3.6
2.8	3.6

Values of $k=0.9$ and 1.8 ; hydrolysis value $=14.8$; $k_{b(15)}=1.62 \cdot 10^{-9}$ —a result rather higher (having regard to difference of temperature) than that, $k_{b(40)}=1.6 \cdot 10^{-9}$, obtained by Allen (*J. Amer. Chem. Soc.*, 1902, 25, 421) by the catalysis method. However, both results show that phenylhydrazine behaves as aniline, in which hydrogen is replaced by an amino-group (which causes an increase of basic value), rather than as hydrazine, in which hydrogen is replaced by a phenyl group.

The separation of base alluded to above prevented any satisfactory results in the case of *p*-bromophenylhydrazine hydrochloride.

Aminophenols and their Derivatives.

o- and *p*-Aminophenol Hydrochlorides.—The following results were obtained for these salts under the conditions of concentration set out and at 15°.

ortho-Salt.		para-Salt.	
I.	II.	III.	IV.
$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
1.2	2.0	0.6	1.1
2.4	4.2	1.1	2.1
3.4	4.2	1.6	3.0
3.4	4.2	2.1	3.0

Values of k for I and II = 1.15 and 2.1, corrected 1.1 and 2.2 respectively; hydrolysis value = 34.4; $k_{b(15)} = 2.18 \times 10^{-10}$; values of k for III and IV = 0.55 and 1.0, corrected 0.53 and 1.06 respectively; hydrolysis value = 8.3; $k_{b(15)} = 6.6 \times 10^{-9}$.

o- and *p*-Anisidine Hydrochlorides.—The results obtained with solutions of these salts, prepared from the respective bases and hydrochloric acid, are given below :

ortho-Salt (temp. 15°).		para-Salt (temp. 17°).	
I.	II.	III.	IV.
$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
1.2	2.7	0.5	1.05
2.5	5.2	1.0	2.1
3.8	7.7	1.6	3.1
5.2	7.7	2.2	4.2
6.4	7.7	2.9	5.2
7.6	7.7	2.9	5.2

Values of k for I and II = 1.25 and 2.55, corrected 1.26 and 2.53 respectively; hydrolysis value = 39.5; $k_{b(15)} = 1.9 \times 10^{-10}$; values of k for III and IV = 0.55 and 1.05; hydrolysis value = 8.4; $k_{b(17)} = 5.7 \times 10^{-9}$.

o- and *p*-Phenetidine Hydrochlorides.—The results obtained are given below :

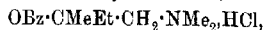
ortho-Salt (temp. 20°).		para-Salt (temp. 15°).	
I.	II.	III.	IV.
$V=4 \times 10^4$.	$V=2 \times 10^4$.	$V=2 \times 10^4$.	$V=1 \times 10^4$.
1.2	2.4	0.8	1.8
2.2	4.3	1.8	3.5
3.1	6.5	2.8	3.5
4.1	8.5	2.8	3.5

Values of k for I and II = 1.05 and 2.2, corrected 1.07 and 2.15 respectively; hydrolysis value = 33.6; $k_{b(20)} = 4.64 \times 10^{-10}$; values of k for III and IV = 0.9 and 1.8; hydrolysis value = 14, $k_{b(15)} = 2.15 \times 10^{-9}$.

Aminobenzoyl Alcohols of the Type $\text{OBz}\cdot\text{CMeR}\cdot\text{CH}_2\cdot\text{NMe}_2$

These alcohols were originally prepared by Fournau (*Compt. rend.*, 1904, 138, 766) by the action of a secondary or tertiary amine on Tiffeneau's chlorohydrins, $\text{OH}\cdot\text{CMeR}\cdot\text{CH}_2\text{Cl}$; the hydrochlorides of their benzoyl derivatives crystallise well. Such compounds belong rather to the aliphatic series, but, since the date of my last communication, MM. Poulenc Frères, of Paris, have kindly presented me with fine, crystalline samples for the purpose of investigation, and I take this opportunity of tendering my thanks to this firm for their courtesy and generosity.

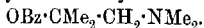
The most important of these compounds, (I) methylethyldimethylaminomethylcarbinol benzoate hydrochloride,



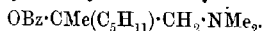
commercially known under the name of stovaine, is now largely used instead of cocaine for hypodermic injections to produce local anaesthesia and other purposes, as being less toxic and safer to administer. It is well known that the base is precipitated from aqueous solutions even by very dilute solutions of borax, the soda liberated by hydrolysis being sufficient to upset the equilibrium between the base and hydrochloric acid. Aqueous solutions are also decomposed when heated in a soda-glass vessel in an autoclave, the course of events being probably that the water dissolves out sodium silicate, which consists largely, in aqueous solutions, of free soda and silicic acid (Kohlrausch, *Wied. Ann.*, 1892, 47, 756), and then the former combines with the hydrochloric acid.

Besides stovaine, the hydrochlorides of the following bases were examined.

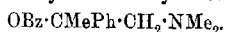
II. Dimethyldimethylaminomethylcarbinol benzoate,



III. Methylisoamyldimethylaminomethylcarbinol benzoate,



IV. Phenylmethyldimethylaminomethylcarbinol benzoate,



All these salts in *N*/20 or *N*/100 solution showed no trace of hydrolysis by the methyl-orange method; hence their basic constant is greater than 1×10^{-7} , which is rather remarkable having regard to the presence of the benzoyl grouping.

A description of the method adopted for differentiating between the basic values of these several salts, and its applicability in other cases, is deferred to a subsequent communication.

(2) *Diacidic Bases.*

Phenylenediamine Hydrochlorides.—These compounds have previously been examined by Bredig (*loc. cit.*) by the electric conductivity method; the writer pointed out that they all gave a strongly acid reaction with methyl-orange, and arrived at the general conclusion that they were hydrolysed to a greater or less degree into the monohydrochloride and hydrochloric acid (as hydrazine hydrochloride). It was thought advisable to make determinations, not only with the *N*/20 solutions diluted directly, but also with the same solutions heated in a thermostat for three hours at 60°, and then subsequently diluted (series marked *T*). But although further information was obtained by the double method, yet the difficulty was experienced that, in the process of heating, the solutions turned a yellow to yellowish-red colour, the change being most marked in the para-derivative, less in the meta-, and inappreciable in the ortho-derivative. The probable explanation is that traces of safranine compounds were formed (compare Witt, *Ber.*, 1883, 16, 472, etc.), the conditions being favourable, namely, the presence of hydrochloric acid liberated by hydrolysis and of oxygen, whether dissolved in the solution or absorbed from the superincumbent atmosphere. The same remarks also apply to the tolylenediamine hydrochlorides, considered in the sequel.

o-Phenylenediamine Hydrochloride.—Three series of experiments were conducted, with results as under:

Series I. Series II (T). Series III.			Series I. Series II (T). Series III.		
$V=1 \times 10^4$. $V=8 \times 10^4$. $V=4 \times 10^4$.			$V=8 \times 10^4$. $V=8 \times 10^4$. $V=4 \times 10^4$.		
0.6	0.8	1.3	4.2	4.9	5.8
1.2	1.5	2.8	5.0	5.6	5.8
2.0	2.3	4.3	5.0	6.4	5.8
2.7	3.1	5.8	5.0	8.3	5.8
3.5	3.9	5.8			

Values of k for series I and III = 0.7 and 1.45, corrected 0.71 and 1.42; hydrolysis value = 44.4; for series III, $k=0.8$; hydrolysis value = 50.0.

m-Phenylenediamine Hydrochloride.—Five series of experiments were conducted, namely, three with one and two with another sample of the salt, purchased at a different time; the results of only three are given, and the differences in values obtained with the two samples were within the limits of experimental error:

Series I. Series II. Series III.			Series I. Series II. Series III.		
$V=8 \times 10^4$. $V=4 \times 10^4$. $V=4 \times 10^4$ (T).			$V=8 \times 10^4$. $V=4 \times 10^4$. $V=4 \times 10^4$ (T).		
0.8	1.4	1.6	3.7	7.0	7.6
1.5	2.6	2.9	4.4	8.5	8.9
2.3	4.0	4.4	5.2	8.5	10.7
3.0	5.5	6.3			

Values of k for series I and II = 0.75 and 1.40, corrected 0.72 and 1.45; hydrolysis value = 45.3; for III, k = 1.55; hydrolysis value = 48.4.

p-Phenylenediamine Hydrochloride.—The results of three series of experiments are given below:

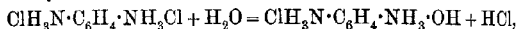
Series I.	Series II.	Series III.	Series I.	Series II.	Series III.
$V = 8 \times 10^4$.	$V = 4 \times 10^4$.	$V = 4 \times 10^4 (T)$.	$V = 8 \times 10^4$.	$V = 4 \times 10^4$.	$V = 4 \times 10^4 (T)$.
0.6	1.3	1.5	4.5	8.4	7.6
1.3	2.9	3.0	5.1	9.9	7.6
2.1	4.3	4.5	5.7	9.9	7.6*
2.8	5.7	6.2	6.4	9.9	7.6
3.7	7.0	7.6	8.0	9.9	7.6

Values of k for series I and II = 0.7 and 1.4; hydrolysis value = 43.7; for series III, k = 1.5; hydrolysis value = 47.0.

In all the above sets of experiments with the isomeric phenylenediamine hydrochlorides a slight tendency for the differences, $y' - y$, to increase was observed, due probably to a trace of hydrolysis of the monohydrochloride; but as the point has frequently been alluded to it is unnecessary to dwell further on it. The results for hydrolysis are compared in the following table:

	x .	$x (T)$.
<i>o</i> -Derivative.....	44.4	50.0
<i>m</i> -Derivative	45.3*	48.4
<i>p</i> -Derivative	43.7	47.0

It thus appears that the phenylenediamine hydrochlorides are, under the conditions of the experiment, hydrolysed nearly completely into the monohydrochloride and hydrochloric acid, thus:



and that the order of affinity of bases is $p > m > o$, the result marked * being an exception not readily explained; it was repeated with two different samples at two different times with concordant results.

Diaminophenol Hydrochloride.

As solutions of this substance commenced to turn yellow shortly after making up, the operations of dilution and examination were conducted as quickly as possible; the following results were obtained in two series:

Series I.	Series II.	Series I.	Series II.
$V = 8 \times 10^4$.	$V = 4 \times 10^4$.	$V = 8 \times 10^4$.	$V = 4 \times 10^4$.
0.7	1.4	4.5	9.2
1.4	2.9	5.2	9.2
2.2	4.4	6.0	9.2
3.0	5.9	6.8	9.2
3.8	7.5		

Values of k for series I and II = 0.75 and 1.50; hydrolysis value = 47.0.

The introduction of the hydroxyl group into an *o*-diaminobenzene appears to cause a slight increase in the hydrolysis value; otherwise the above remarks are equally applicable.

Phenylenetetramethyldiamine Hydrochloride.—Solutions of this substance were almost impossible to work with, owing to the rapidity with which they assumed a bluish-violet colour, doubtless from the formation of a trace of some induline derivative; the results of one series are given:

$V=8 \times 10^4$.	$V=8 \times 10^4$.
0.8	3.1
1.5	3.9
2.2	3.9

$k=0.8$; hydrolysis value = 50.0.

Tolylenediamine Hydrochlorides.—The 2:4-, 2:5-, and 3:4-derivatives were examined. The results are given below:

2:4-Derivative.

Series I. $V=8 \times 10^4$.	Series II. $V=8 \times 10^4 (T)$.	Series III. $V=4 \times 10^4$.	Series I. $V=8 \times 10^4$.	Series II. $V=8 \times 10^4 (T)$.	Series III. $V=4 \times 10^4$.
0.6	0.7	1.5	4.4	4.9	9.7
1.2	1.5	3.2	5.2	5.7	11.4
2.1	2.3	4.8	6.0	6.6	11.4
2.7	3.1	6.3	6.7	6.6	11.4
3.5	4.0	8.2	7.6	6.6	11.4

Values of k for series I and III = 0.75 and 1.6, corrected 0.78 and 1.55; hydrolysis value = 48.4; for series II, $k=1.6$; hydrolysis value = 50.0.

2:5-Derivative.

Series I. $V=8 \times 10^4$.	Series II. $V=4 \times 10^4$.	Series I. $V=8 \times 10^4$.	Series II. $V=4 \times 10^4$.
0.7	1.5	4.2	8.7
1.5	2.9	5.0	10.3
2.1	4.3	5.9	10.3
2.8	5.7	6.7	10.3
3.6	7.2	7.3	10.3

Values of $k=0.7$ and 1.45, corrected 0.71 and 1.42; hydrolysis value = 44.4.

Solutions of this substance, when heated to 60°, became too highly coloured for investigation.

3:4-Derivative.

Series I. $V=8 \times 10^4$.	Series II. $V=8 \times 10^4 (T)$.	Series III. $V=4 \times 10^4$.	Series I. $V=8 \times 10^4$.	Series II. $V=8 \times 10^4 (T)$.	Series III. $V=4 \times 10^4$.
0.8	0.8	1.4	3.9	4.1	7.1
1.5	1.6	2.7	4.6	5.0	8.5
2.3	2.4	4.2	5.4	5.9	10.2
3.1	3.2	5.6			

Values of k for series I and III = 0.75 and 1.40, corrected 0.72 and 1.45 respectively; hydrolysis value = 45.0; for series III, $k = 0.8$; hydrolysis value = 50.0.

It will be evident from the above results that the behaviour of the tolylenediamines is completely analogous to that of the phenylenediamines, in that they are hydrolysed nearly completely into the monohydrochloride and hydrochloric acid, and the change is complete if the solutions are previously heated. Bredig (*loc. cit.*) examined the 2:4 derivative, and his results at dilution $V = 2048$ point to a similar conclusion.

Benzidine Hydrochloride.—Owing to the sparing solubility of this salt, the usual method of procedure was slightly altered; an $N/40$ instead of an $N/20$ original solution was prepared, and this was subsequently diluted. The results obtained are given below (temperature 16°).

$V = 8 \times 10^4$	$V = 4 \times 10^4$	$V = 8 \times 10^4$	$V = 4 \times 10^4$
0.8	1.4	3.9	7.4
1.7	2.8	4.6	8.8
2.4	4.4	4.6	10.4
3.1	6.0		

Values of $k = 0.75$ and 1.5 ; hydrolysis value = 46.6.

The behaviour of benzidine hydrochloride is thus perfectly analogous to that of the diaminobenzenes; my results are not in this respect in accordance with those of Bredig (*loc. cit.*), who concluded from his measurements that the salt was only slightly hydrolysed.

Dibenzylamine and *diphenylamine* hydrochlorides were not sufficiently soluble, or gave the insoluble base on hydrolysis; the methyl-orange method could not therefore be applied.

Naphthylamines.

α - and β -Naphthylamine Hydrochlorides.—My experience in obtaining these compounds in a sufficient state of purity for examination was similar to that noted by Lellmann (*Annalen*, 1891, 263, 297), and it appeared that the impurities present, of whatever nature, were of an order of value less than that deducible by a determination of the chlorine contents. The criterion of purity finally adopted was that the $N/20$ solutions of the recrystallised salts should withstand heating for three hours at 60° without becoming discoloured, or showing only traces of fluorescence. The values of k , obtained were in both cases higher than those obtained by Farmer and Warth (*loc. cit.*), who give no details as to any special precautions adopted in the preparation of the salts examined.

α -Salt (temp. 15°).

I.	II.
$V=4 \times 10^4$.	$V=2 \times 10^4$.
1.2	2.0
2.2	4.0
3.1	8.0
4.3	8.1
5.4	10.4
6.7	10.4
8.0	10.4
9.2	10.4
10.4	10.4

β -Salt (temp. 15°).

III.	IV.
$V=4 \times 10^4$.	$V=2 \times 10^4$.
1.1	1.9
2.1	3.6
3.1	5.5
4.1	5.5
5.0	5.5
5.0	5.5
5.0	5.5
5.0	5.5
5.0	5.5

Values of k for I and II = 1.15 and 2.0, corrected 1.08 and 2.05 respectively; hydrolysis value = 33.4; $k_{b(15)} = 2.8 \cdot 10^{10}$ (Farmer and Warth found $k_{b(25)} = 0.99 \cdot 10^{-10}$); values of k for III and IV = 1.0 and 1.8, corrected 0.95 and 1.9 respectively; hydrolysis value = 29.7, $k_{b(15)} = 3.9 \cdot 10^{-10}$ (Farmer and Warth found $k_{b(25)} = 2.0 \cdot 10^{-10}$). The results obtained by these writers and myself concur in showing that β -naphthylamine is a stronger base than α -naphthylamine.

Pyridine Bases.

Pyridine Hydrochloride.—It was found in the case of this salt that equilibrium of the system water-acid-base-salt was attained after a longer interval (two or three days) than in all other cases hitherto examined. Three solutions were examined at 13°, with the following results:

$V=4 \times 10^4$. $V=3 \times 10^4$. $V=2 \times 10^4$.			$V=4 \times 10^4$. $V=3 \times 10^4$. $V=2 \times 10^4$.		
0.9	1.2	1.8	2.5	3.6	6.7
1.7	2.4	3.5	2.5	3.6	8.5
2.5	3.6	5.2			

Values of $k = 0.85$, 1.2, and 1.7, corrected 0.86, 1.18, and 1.73 respectively; hydrolysis value = 26.1; $k_{b(15)} (at V = 2 \times 10^4) = 1.06 \cdot 10^{-9}$. Lundén (*Meddel. K. Vetensk. Nobelinst.*, 1907, 1, No. 8 *et seq.*) has determined the value of k_b for pyridine at different temperatures by the electric conductivities of the hydrochloride and acetate. The value given above is identical with that found by this author at 10°, but otherwise is more nearly concordant with it than with those obtained by Constam and White (*Amer. Chem. J.*, 1903, 29, 36) and Goldschmidt (*Zeitsch. physikal. Chem.*, 1899, 29, 89). In this connexion I may be allowed to remark that my determinations were made some months before Herr Lundén was courteous enough to send me his publications, from which I have derived considerable assistance; the convenience of dividing the bases into open-chain and cyclic compounds has been the sole cause of my delay.

Piperidine Hydrochloride.—No trace of hydrolysis could be detected

even in most concentrated solutions of this salt, a result to be expected, as Bredig (*loc. cit.*) found for piperidine a higher basic constant, $k_b = 1.58 \cdot 10^{-2}$, than that of any other nitrogen-containing base; its heat of neutralisation with hydrochloric acid is practically equal to that of sodium hydroxide. Piperidine has probably the constitution of a hexamethylene, in which one CH_2 group is replaced by the NH group.

Conine (Propylpiperidine) Hydrochloride.—The result obtained was precisely similar to that of piperidine.

The stability of these hydropyridine hydrochlorides is otherwise of general importance, having regard to the number of natural alkaloids which are derived from a hydropyridine ring.

α -Picoline Hydrochloride.—Prepared in solution by dissolving a weighed quantity of the base in the required volume of $N/20$ hydrochloric acid. Only a few results could be obtained, as the limit of the methyl-orange method was almost reached. The hydrolysis value found at 15° was 1.9, giving for the dilution used a value, $k_{b(15)} = 5.4 \cdot 10^{-8}$, which can only be regarded as an approximation, although probably not widely removed from the true value as intermediate between those of pyridine, $k_b = 1.3 \cdot 10^{-9}$, and trimethylpyridine, $k_b = 1.42 \cdot 10^{-7}$, at the same temperature.

Quinoline Bases.

Quinoline Hydrochloride.—The results obtained at 15° for two concentrations are given below:

$V = 2 \times 10^4$	$V = 1 \times 10^4$	$V = 2 \times 10^4$	$V = 1 \times 10^4$
1.0	1.9	5.9	—
2.0	4.2	6.8	—
3.1	6.0	7.8	—
4.1	7.8	8.9	—
5.0	—		

Values of $k = 1.0$ and 2.0 respectively; hydrolysis value = 15.6 ; hence $k_{b(15)} = 1.63 \cdot 10^{-9}$; this last value is in fair accordance with that found, $k_{b(60)} = 7.4 \cdot 10^{-9}$, by Walker and Aston (*Trans.*, 1895, 67, 576) by the inversion of sucrose method, having regard to the difference of temperature of the two sets of observations.

Tetrahydroquinoline Hydrochloride.—No hydrolysis could be detected even in concentrated solutions; the behaviour of this substance is thus perfectly analogous to the hydro-derivatives of pyridine.

2-Hydroxyquinoline (Carbostyryl) Hydrochloride.—A solution of this salt was obtained by dissolving a weighed quantity of the base in $N/20$ hydrochloric acid. The following results were obtained at 16° with two concentrations:

$V = 2 \times 10^4$	$V = 1 \times 10^4$	$V = 2 \times 10^4$	$V = 1 \times 10^4$
1.1	2.1	3.1	4.0
2.0	4.0	4.2	4.0

Values of $k = 1.05$ and 2.08 , corrected 1.03 and 2.07 ; hydrolysis value $= 16.6$; hence $k_{b(18)} = 1.94 \cdot 10^{-9}$.

The general behaviour of hydroxyquinoline as compared with quinoline is similar to that of *p*-aminophenol as compared with aniline, in that the introduction of the hydroxyl group, although not causing any appreciable difference in the affinity constant, yet produces the effect that all reaction with the methyl-orange sooner comes to an end.

2-Methylquinoline Hydrochloride.—A solution of this salt was prepared as above, and two concentrations were examined at 14° :

$V = 2 \times 10^4$	$V = 1 \times 10^4$	$V = 2 \times 10^4$	$V = 1 \times 10^4$
0.7	1.3	2.5	5.0
1.3	2.5	2.5	6.2
1.9	3.8	2.5	7.3

Values of $k = 0.65$ and 1.28 ; hydrolysis value $= 9.9$; therefore $k_{b(14)} = 3.63 \cdot 10^{-9}$; this base is therefore slightly stronger than quinoline.

isoQuinoline Hydrochloride.—A solution of this salt was prepared as above, and two concentrations were examined at 15° :

$V = 4 \times 10^4$	$V = 2 \times 10^4$	$V = 4 \times 10^4$	$V = 2 \times 10^4$
0.9	1.9	4.8	9.9
1.8	3.9	5.7	—
2.9	6.0	6.7	—
3.8	8.0	—	—

Values of $k = 0.95$ and 2.0 ; corrected 0.97 and 1.95 ; hydrolysis value $= 30.2$; hence $k_{b(15)} = 3.62 \cdot 10^{-10}$; this base is therefore weaker than quinoline.

Acridines.—I have to express my thanks to Prof. Senier for kindly supplying me with beautiful specimens of these bases; my only regret is that they proved to be insufficiently soluble in hydrochloric acid of the concentration required for this investigation.

Section II.—Mutual Relationship.

(1) *Effect of Position on Isomerism*.—The order of the affinity constants of the isomeric derivatives of benzene is invariably $p > m > o$, or, in other words, the para-position presents a case of steric hindrance, but the ortho a case of steric furtherance; this generalisation is precisely analogous to that arrived at in the case of acids by Ostwald (*Zeisch. physikal. Chem.*, 1889, 3, 170 *et seq.*) and myself by the methyl-orange method (*ibid.*, 1906, 57, 147 *et seq.*). The results for k_b are compared in the following table:

	<i>p</i> -Base.	<i>m</i> -Base.	<i>o</i> -Base.
Toluidines	$4.5 \cdot 10^{-10}$	$3.5 \cdot 10^{-10}$	$1.52 \cdot 10^{-10}$
Dimethyltoluidines ...	$6.36 \cdot 10^{-9}$	—	$3.08 \cdot 10^{-9}$
Chloroanilines	$1.24 \cdot 10^{-11}$	$6.58 \cdot 10^{-12}$	$9.16 \cdot 10^{-12}$
Bromoanilines	$2.07 \cdot 10^{-10}$	$9.5 \cdot 10^{-11}$	—
Aminophenols	$6.6 \cdot 10^{-9}$	—	$2.18 \cdot 10^{-10}$
Anisidines	$5.7 \cdot 10^{-9}$	—	$1.9 \cdot 10^{-11}$
Phenetidines	$2.15 \cdot 10^{-9}$	—	$4.64 \cdot 10^{-10}$

In certain cases there is the same numerical ratio between corresponding isomerides:

thus *m*-chloroaniline: *p*-chloroaniline = $6.58 \cdot 10^{-12} : 12.4 \cdot 10^{-12} = 1:1.9$,
 and *m*-bromoaniline: *p*-bromoaniline = $9.5 \cdot 10^{-11} : 20.7 \cdot 10^{-11} = 1:2.2$,
 also *p*-aminophenol: *o*-aminophenol = $6.6 \cdot 10^{-9} : 0.22 \cdot 10^{-9} = 30:1$,
 and *p*-anisidine: *o*-anisidine = $5.69 \cdot 10^{-9} : 0.19 \cdot 10^{-9} = 285:1$.

(2) *Effect of Substitution.*—(a) The substitution of hydrogen by bromine produces a less effect on the affinity constant than that of hydrogen by chlorine, a conclusion also analogous to that arrived at by Ostwald in the case of the acids.

(b) The substitution of hydrogen by the hydroxyl, methoxyl, or ethoxyl groups in the para-position increases the affinity constant, but in the ortho-position produces but little effect.

(c) As regards the substitution of hydrogen by hydrocarbon groups, whether in the amino- or hydrocarbon residue, it appears difficult to draw any very definite conclusion, although generally the effect is to increase the basic constant, but there are certain exceptions. Bredig arrived at a similar uncertain conclusion.

(d) The effect of substitution of hydrogen by an amino-group when attached to a carbon atom decreases the affinity constant, the diamines being hydrolysed uniformly to 50 per cent., but an amino-group attached to a nitrogen atom produces an opposite effect, for example, the case of phenylhydrazine as compared with aniline, which is analogous to that of semicarbazide with carbamide and of amino-guanidine with guanidine alluded to in my previous communication.

(e) The addition of hydrogen to the pyridine and quinoline bases produces a very great increase in the affinity constant, although, as pointed out above, such additive compounds may possess a different constitution, not readily expressible by structural formulae.

Section III.—Temperature-Coefficients.

Kohlrausch and Heydweiller (*Wied. Ann.*, 1894, 53, 209), from their determinations of the electric conductivity of the purest water at different temperatures (and hence the temperature-coefficient referred to 18°), deduced by means of the van't Hoff gas law a general equation:

$$C = 0.03373 \cdot 10^{-22500/T_2} \quad (2),$$

or the ionic concentration $C_{(H)}$ or $C_{(OH)}$ per litre. By two independent methods of calculation, they arrived at the values given in the first line of the succeeding table; the values of $k_w = (C_H)^2$ (since $C_H = C_{(OH)}$) calculated therefrom are given in the second column (compare Abegg, *Sammlung Chem. und Chem.-techn., Vorträge*, 1903, 8, 242); in the third column, certain results obtained by Lundén (*Meddel. K. Vetensk. Nobelinst.*, 1907, 2, 16) are added.

	0.	10.	15.	18.	26.	34.	42.	50.
ionic concentration/litre 10^{-7} .	0.35	0.56	—	0.8	1.09	1.47	1.93	2.48
$k_w^{(1)} 10^{-14}$	0.12	0.31	0.49	0.64	1.2	2.15	3.7	6.15
"	—	0.31	0.46	—	1.05 ⁽²⁰⁾	—	2.94 ⁽⁴⁰⁾	5.17

Abegg remarks upon the rapid increase of ionic dissociation of water with rise of temperature.

It appears, however, that the above values can be directly calculated by the expression originally proposed by Harcourt and Esson (*Phil. Trans.*, 1895, 186, A, 861) for the relation of chemical change for temperature, namely:

$$\frac{dk}{k} \bigg/ \frac{dT}{T} = m \quad \dots \quad (3),$$

or on integration,

$$k_T/k_T = (T'/T)^m \quad \dots \quad (4),$$

which may be written in the simple form

$$\log k - \log k' = m(\log T' - \log T) \quad \dots \quad (5).$$

In the above equations 5.3—5, k represents either C_H or k_w , T absolute temperature, and m a factor.

If the value of C_H at 0 is taken as $0.37 \cdot 10^{-7}$ and $m = 11$, and $k_w = 0.14 \cdot 10^{-14}$ and $m = 22$ (or preferably 22.5), then the values at different temperatures appear as under:

	0.	10.	15.	18.	26.	34.	42.	50.	60.
$C_H \cdot 10^{-7}$	0.37	0.56	0.68	0.76	1.01	1.36	2.08	2.49	3.5
$k_w \cdot 10^{-14}$...	0.14	0.31	0.47	0.58	1.02	1.85	4.3	6.25	12.25

The agreement between the numbers in the two tables is very satisfactory. Hence, therefore, the logarithmic increase of ionic dissociation due to increase of temperature varies as the logarithmic increment of absolute temperature, or, to put the matter in another way, ionic dissociation of water as affected by temperature cannot be differentiated from other chemical changes, and consequently there is nothing remarkable in the increase of such dissociation with rise of temperature. The graph of $\log k'/\log k$ in terms of $\log T'/\log T$ is a straight line, and this may serve as a convenient method of ascertaining the value of k_w for any temperature.

But from the Arrhenius' equation: $k_h/k_{h0} = (1-x)V/x^2$, if k_{h0} is an

experimental function of temperature, k_p will likewise be of the same order. In the following table, the results obtained by various observers are compared with those calculated by formula (4), the value of the function m being deduced; most of the observations are referred to a basis $T=283$.

The results are taken from the writings of Bredig, Walker, Lundén, myself, and others; in cases in which different workers have obtained slightly different results at the same temperature by applying different methods, the letters (A), (B), etc. are used,

Temperature.	10.	15.	25.	40.	50.	60.	
Ammonia, 10^{-5}	{ 1.63	1.71	1.87	1.98	—	—	Found
$m=2$	{ 1.63	1.69	1.81	1.99	—	—	Calc.
Trimethylpyridine,	{ 1.22	1.42	2.05	3.05	3.75	—	Found
10^{-7} , $m=9$	{ 1.22	1.42	1.94	3.01	4.00	—	Calc.
	{ 1.06	1.41(A)	2.38(A)	4.25(A)	6.19(A)	8.5(D)	Found
Pyridine, 10^{-9}		1.06(B)	2.00(C)				
$m=14$	{ 1.00	1.27	2.06	4.08	6.19	9.71	Calc.
	{ 2.6 (12°)	3.66(A)	4.57(A)	7.56(A)	—	17.1(D)	Found
Aniline, 10^{-10} , $m=12$		3.2 (B)	4.6 (C)	8.1 (D)			
	{ 2.71(12°)	3.07	4.64	8.34	—	17.7	Calc.
<i>o</i> -Toluidine, 10^{-10} ,	{ 1.5(12°)(A)	—	3.2 (B)	—	—	10.9(C)	Found
$m=13$	{ 1.5(12°)	—	2.6	—	—	10.9	Calc.
<i>p</i> -Toluidine, 10^{-9} ,	{ —	0.45(A)	1.4 (B)	—	—	3.61(C)	Found
$m=15$	{ [0.3]	0.4	0.69	—	—	3.42	Calc.

The differences between the observed and calculated results are with one or two exceptions very small, especially having regard to the fact that the methods applied were in some cases very widely different. In the case of very weak bases of the order of value k_b 10^{-11} to 10^{-12} , concordance is less satisfactory, but as the determination of the hydrolysis of 90 per cent., or thereabouts, may amount to 5 per cent. or even more discrepancies, especially at the higher temperature, cannot but be expected.

It would follow, therefore, that if both k_b and k_w are exponential functions of absolute temperature, then in the thermodynamic equation

$$\phi = RT^2 \cdot 2.3026 \log^{10} k/dT \quad \therefore \dots (6),$$

ϕ = quantity of heat, k = constant of equilibrium, and, since $R=1.985$ of the unit of work in the gram-caloric (compare Nernst, *Zeitsch. Elektrochem.*, 1904, 10, 621)

$$\phi = 4.571 T^2 \log^{10} k/dT \quad \dots (6 bis),$$

it follows that the heats of neutralisation (by the substitution $k=k_w/k_b$) and the heats of dissociation (by the substitution $k=k_b$) are exponential functions of absolute temperature. These can be directly calculated for any temperature without having recourse to mean values or the application of empirical equations:

$$k = 1 + at + bt^2, \text{ etc.} \quad \dots (7),$$

or other arithmetical devices. I refrain from giving such calculations of heats of neutralisation and of dissociation, as I am aware that other writers have been engaged upon them; my object is to point out a simple and possibly more rational method. I am indebted to Professor Wm. Esson, of the University of Oxford, for assistance in this section.

Summary.

(i) It is shown by a number of determinations that the methyl-orange method gives results which are concordant within the limits of experimental error with those obtained by the catalysis of methyl acetate, inversion of sucrose, and electric conductivity methods. If the last-named give accurate results, then the same equally applies to the first. It is, of course, possible that the affinity constants of the bases may at some future date be revised, as they are deduced from the values of k_{sp} or C_{sp} , which at the present time are mainly based on the determinations at different temperatures of the electric conductivity of the purest water obtained by Kohlrausch, and these in their turn are dependent on the evaluation of the ohm.

(ii) Determinations are given of the hydrolysis and affinity constant values of some fifty bases derived from cyclic formulæ; the mutual relationship and effects produced by the substitution of one or more atomic proportions of hydrogen, whether in the nucleus or side-chain, by various elements or groups are discussed.

(iii) The variation of the several constants with temperature is dealt with, and it is shown that in cases in which determinations of these constants have been made at different temperatures by the same or different trustworthy methods, the experimental results are in accordance with those calculated by Esson's formula $k_{T_1}/k_T = (T_1/T)^m$. Thus ionic dissociation, whether of water or electrolytes, cannot be differentiated in respect of temperature from any other change, and is generally regarded as of a wholly chemical nature. The importance of this formula for the purpose of other calculations is also alluded to.

Finally, I have again to express my obligations to various friends, who have rendered assistance by verbal or written suggestions, or supplied necessary materials.

Addendum.

Since the above paper was written, a communication has been received from Herr Lundén, entitled "Influence of temperature on the internal energy and free energy of electrolytic dissociations of acids and weak bases" (*Meddel. K. Vetensk. Nobelinst.*, 1907, I, No. 12). In this paper, certain formulæ are discussed with the

purpose of referring affinity values, as influenced by temperature, to thermodynamic laws. The equation given is:

$$\log_{nat} k_{T_1} - \log_{nat} k_{T_2} = \frac{T_2}{T_1} \int_{T_1}^{T_2} \frac{(U/RT^2) dT}{T} \quad (1a),$$

in which U = internal energy, also a function of T with two constants, namely, $U = f(ab^T)$, and the simplest, as also that most in accordance with observations, is $U = a + bT$.

It will be noticed that the left-hand side of the above equation (1a) differs from mine, $\log_{10} k_{T_1} - \log_{10} k_{T_2}$, given in the text, only in degree, whilst the right-hand side refers the difference of affinity values to thermodynamic laws, and thus takes matters one step further than that given in the text, namely, $m(\log_{10} T_1 - \log_{10} T_2)$.

As I was aware from private communications that Herr Lunden intended to take up the subject of the heats of dissociation and of neutralisation with reference to affinity values, it appeared equitable that this part of the subject should be left to him.

V. H. V.

CCXIV.—Organic Derivatives of Arsenic. Part I. *Dicamphorylarsinic Acid.*

By GILBERT T. MORGAN and FRANCES M. G. MICKLETHWAIT.

THE organic derivatives of arsenic have of late received increased attention owing to the discovery that certain of these substances are of therapeutic value, and, owing to the successful application of sodium *p*-aminophenylarsonate ("atoxyl") in this connexion, many experiments have recently been made with the object of obtaining similarly constituted compounds. In these researches the arsenic has been generally, although not invariably, employed in the form of arsenic acid. Considered as a synthetical agent, arsenic acid has the disadvantage of being hydrated and not very miscible with the ordinary organic media. On the other hand, arsenious chloride is easily obtained in the anhydrous state, and is readily miscible with non-hydroxylic solvents. Moreover, on account of its volatility, any excess of this reagent can often be removed by distillation.

The experiments of Michaelis and his collaborators (*Annalen*, 1892, 270, 140; 1901, 320, 271; 1902, 321, 141) have shown that arsenious chloride may be employed as the vehicle for introducing arsenic into aromatic nuclei.

The authors have examined the action of arsenious chloride on

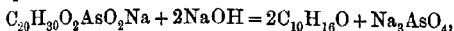
compounds of a very dissimilar type, and in the present communication they wish to record the results obtained in the case of camphor.

This ketone was employed in the form of its very reactive sodium derivative, a substance which was first employed as a synthetical agent by Haller (*Compt. rend.*, 1891, 112, 1490; 1892, 113, 22), and more recently by Forster (*Trans.*, 1901, 79, 987).

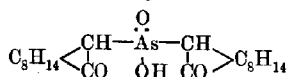
The condensation of arsenious chloride and sodium camphor was effected in dry toluene, and from the product of the reaction caustic alkalis extracted an acidic substance, the composition of which corresponded with the empirical formula $C_{20}H_{31}O_4As$.

Titration with standard caustic alkalis and the formation of a silver salt, $C_{20}H_{30}O_4AsAg$, and a cadmium salt, $C_{40}H_{60}O_8As_2Cd$, indicated that the substance was a monobasic acid, $C_{20}H_{30}O_4AsH^+$.

Further evidence as to the constitution of the acid was gained by a study of the products of its hydrolysis. The acid is not affected by prolonged boiling with aqueous acids or alkalis, but, when fused at a moderate temperature with potassium or sodium hydroxide, camphor (2 mols.) is eliminated and the corresponding alkali arsenate (1 mol.) is produced. This hydrolysis,



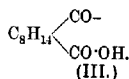
takes place quantitatively, and shows unmistakably that the compound is *dicamphorylarsinic acid* * having the constitution



A further confirmation of this formulation was afforded by the formation of a somewhat unstable *chloride*, $(C_{10}H_{15}O)_2AsO \cdot Cl$, produced by the action of phosphorus pentachloride on the alkali salts of the acid.

The authors wish to reserve for the present the study of the action of arsenious and other inorganic chlorides on organic compounds containing the group $CH_2 \cdot CO$.

* It should be noted that the expression "camphoryl" has been used in the literature to indicate the three following groups:



The authors feel justified in employing "camphoryl" for the first of these radicals, because, in accordance with the terminology usually adopted, the acidic radicle (II) should be denoted by "camphoroyl" or some similar name ending in "oyl." In this respect they follow the ruling already laid down in the case of the groups, $C_6H_5 \cdot CH:CH \cdot CH_2-$ and $C_6H_5 \cdot CH:CH \cdot CO-$, which are indexed in this Journal as *cinnamyl* and *cinnamoyl* respectively.

EXPERIMENTAL.

Seventy-five grams of camphor were dissolved in 200 c.c. of warm toluene, and converted into sodium camphor by the addition of 7.5 grams of sodium. The precipitated sodium derivative, suspended in 200 c.c. of fresh toluene, was slowly treated with 38 grams of arsenious chloride diluted with about twice its bulk of the same solvent, the mixture being thoroughly shaken and cooled. The condensation took place with considerable generation of heat, and a remarkable series of changes occurred during the addition of the chloride. At first the mixture acquired a jelly-like consistence, and gradually assumed a deep crimson hue. This colour slowly faded while the mixture regained its fluidity, until finally it consisted of a yellow, mobile solution with a pulverulent, white precipitate of sodium chloride. After one hour, the mixture was warmed on the steam-bath, and throughout the experiment moisture was excluded.

The mixture was now poured into water, and extracted with hot aqueous sodium hydroxide. The alkaline extract, when cooled and acidified with hydrochloric acid, furnished a brownish-white precipitate, the yield of which was about 10 per cent., calculated on the weight of camphor employed.

This acidic product was crystallised from benzene, when almost colourless crystals were obtained. Further crystallisation from alcohol gave transparent, colourless, highly refractive, obliquely truncated prisms melting with decomposition at 266° . This decomposition point was, however, considerably lowered when the acid was heated for some time at 150° :

0.1300 gave 0.2752 CO_2 and 0.0930 H_2O . $\text{C} = 57.72$; $\text{H} = 7.95$.

0.1418 „ 0.3038 CO_2 „ 0.0982 H_2O . $\text{C} = 58.41$; $\text{H} = 7.69$.

0.5132 „ 0.1984 $\text{Mg}_2\text{As}_2\text{O}_7$. $\text{As} = 18.71$.

$\text{C}_{20}\text{H}_{31}\text{O}_4\text{As}$ requires $\text{C} = 58.53$; $\text{H} = 7.56$; $\text{As} = 18.29$ per cent.

0.8870, titrated with standard sodium hydroxide solution, required 0.08604 NaOH . The monobasic acid, $\text{C}_{20}\text{H}_{30}\text{O}_4\text{AsO}\cdot\text{OH}$, requires 0.08653 NaOH .

0.4890, in 25 c.c. chloroform in a 2-dcm. tube, gave $\alpha_D^{20} + 7.3^{\circ}$; whence $[\alpha]_D^{20} + 186.6^{\circ}$.

Dicamphorylarsinic acid, $(\text{C}_{10}\text{H}_{15}\text{O})_2\text{AsO}\cdot\text{OH}$, is almost insoluble in water or petroleum; it dissolves more readily in benzene, and is freely soluble in chloroform or alcohol. From the last of these solvents it separates in lustrous, colourless crystals. In titrating this sparingly soluble acid, a weighed portion, dissolved in benzene, was shaken up with water containing phenolphthalein, when a sharp end-point was obtained with either $N/2$ -sodium or $N/2$ -potassium hydroxide.

The salts of the alkali metals and ammonium are extremely soluble in water or alcohol. The calcium, strontium, barium, nickel, and cobalt salts are not precipitated in aqueous solutions; the ferric, mercuric, and cupric salts are almost insoluble in water.

Silver dicamphorylarsinate, $(C_{10}H_{15}O)_2AsO \cdot OAg$, was obtained as a white, sparingly soluble precipitate from sodium dicamphorylarsinate and silver nitrate; it was amorphous at first, but slowly became crystalline, this change being accelerated on warming the mixture:

0.1398 gave 0.0287 Ag. $Ag = 20.53$.

$C_{20}H_{30}O_4AgAs$ requires $Ag = 20.88$ per cent.

Cadmium dicamphorylarsinate, $[(C_{10}H_{15}O)_2AsO]_2Cd$, separated as a sparingly soluble, white, crystalline compound on mixing strong aqueous solutions of cadmium chloride and potassium dicamphorylarsinate:

0.4316 gave 0.0570 CdO . $Cd = 11.40$.

$C_{40}H_{60}O_8As_2Cd$ requires $Cd = 12.17$ per cent.

From aqueous solutions of its salts, dicamphorylarsinic acid is set free by acetic acid, but only a very slight precipitate is produced by carbonic acid. Dicamphorylarsinic acid does not yield an oxime on treatment with hydroxylamine in hot aqueous or alcoholic solutions.

Dicamphorylarsinyl chloride, $(C_{10}H_{15}O)_2AsO \cdot Cl$, obtained by the interaction of potassium dicamphorylarsinate and phosphorus pentachloride, separated from chloroform and benzene in colourless crystals melting at 158° :

0.2158 gave 0.0646 $AgCl$. $Cl = 7.41$.

$C_{20}H_{30}O_3ClAs$ requires $Cl = 8.28$ per cent.

This substance is very sensitive to moisture, and is rapidly decomposed on exposure to the atmosphere; its specific rotation, taken in dry chloroform, gave $[\alpha]_D +106^\circ$.

Hydrolysis of the Alkali Dicamphorylarsinates.

Although stable in hot aqueous solutions, the alkali dicamphorylarsinates, when evaporated with excess of caustic alkali until the mixture assumed a syrupy consistence, underwent hydrolysis with the liberation of camphor and the formation of an alkali arsenate. The camphor was evolved quantitatively, and in one experiment 97 per cent. of the calculated amount was collected and identified by its melting point, specific rotation, and conversion into camphoroxime (m. p. 118°). The alkali arsenate was identified by conversion into copper arsenate, and also by precipitating the arsenic acid as magnesium ammonium arsenate.

When heated at 300°, dicamphorylarsinic acid and its alkali salts underwent complete decomposition.

The authors desire to express their thanks to the Government Grant Committee of the Royal Society for a grant which has partly defrayed the expenses of this investigation.

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CCXV.—*Study of the Constitution and Properties of the Rhodanides of Inorganic Radicles. Part I.*

By AUGUSTUS EDWARD DIXON and JOHN TAYLOR.

It is well known that certain more or less electronegative atomic complexes, for example, CNS, CNO, CN, or NO₂, may yield with a given electropositive organic radicle in each case two isomeric compounds, the isomerism of which is conditioned by the mode of attachment of the particular atomic complex. Thus the chemical differences between the two forms of ethyl rhodanide, C₂H₅(CNS), are satisfactorily explained by the constitutional formulæ Et·S·C≡N and Et·N·C≡S respectively.

Our knowledge regarding the combinations of distinctly electronegative organic radicles with such electronegative atomic complexes is confined in effect to the series of compounds derived from carboxylic acids. Here no case of isomerism has yet been observed, the same being true with respect to the combinations of hydrogen and of the metals with the atomic complexes just mentioned; for instance, no isomeride is known of hydrocyanic acid or of potassium thiocyanate. Ammonium thiocyanate, it is true, has the same molecular composition as thiourea, NH₂·C(SH)·NH₂, but the production of the strict isomeride, namely, ammonium isothiocyanate, NH₄·N·C≡S, still remains to be accomplished.

Rhodanides of the carboxylic acids may be prepared by the action of a suitable metallic thiocyanate on the corresponding acylogen, for instance:

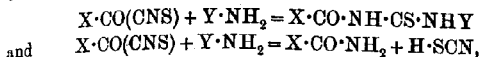
$$2\text{CH}_3\cdot\text{COCl} + \text{Pb}(\text{SCN})_2 = \text{PbCl}_2 + 2\text{CH}_3\cdot\text{CO}(\text{CNS})$$

(Miquel, *Ann. chim. phys.*, 1877, [v], 11, 295). Of such compounds, thirty or so have now been prepared.

Since by direct union with an alcohol these rhodanides yield the corresponding acyl-substituted thiocarbamates, and with primary or

secondary nitrogen bases the corresponding thiocarbimides or thioureas, they must presumably be classed as thiocarbimides, $R \cdot NCS$.

Not infrequently, however, the two forms of change, namely, thiocarbimide union and thiocyanic decomposition :

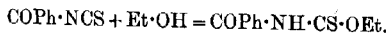


occur concurrently, the relative extents being determined by various external conditions (Doran, *Trans.*, 1905, **87**, 331; Dixon and Hawthorne, *ibid.*, 1906, **89**, 468), from which it might be imagined that the rhodanide constitutes an equilibrium mixture of thiocyanate and thiocarbimide in ratios determined by these conditions.

This, however, is not the case, for the molecular refraction of acetyl rhodanide is sensibly constant between 13° and 75° , and the refraction value of its CNS-group is equal to that determined by experiment for various thiocarbimides, whilst differing widely from that found for aliphatic thiocyanates (Hawthorne, *Trans.*, 1906, **89**, 536).

Furthermore, it has been observed that when α -acetyl-*bb*-diphenylthiocarbimide, $CH_3 \cdot CO \cdot NH \cdot CS \cdot N(C_6H_5)_2$, is caused to dissociate into its constituents, namely, diphenylamine and acetylthiocarbimide, $CH_3 \cdot CO \cdot NCS$, the latter is identical with ordinary acetyl "thiocyanate," this chemical evidence going to confirm the conclusion already reached by Hawthorne on purely physical grounds. Careful examination of the properties, both physical and chemical, of several different acyl rhodanides has served to corroborate the view that, although these compounds may be caused to yield thiocyanic acid by hydrolysis, they are nevertheless thiocarbimide in constitution (Dixon and Taylor, *Trans.*, 1908, **93**, 691).

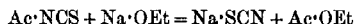
Whether an acyl thiocyanate is formed, even temporarily, from a strongly acid chloride and a metallic thiocyanate seems doubtful; thus Miquel observed (*loc. cit.*) that when benzoyl chloride is added to alcoholic potassium thiocyanate, maintained at 0° , the organic product of the interaction unites with the alcohol to form a substituted thiourethane, and hence, presumably, consists of benzoylthiocarbimide :



Why acyl radicles should be so reluctant to enter into thiocyanic combination it cannot at present be stated with certainty. Disposing causes, however, may be suggested; for instance, the similarity in electrical character of the two constituent groups. Even amongst undoubted thiocarbimides of this class, the union between the acyl and the rhodanic complex may very readily be sundered; thus, so well marked a thiocarbimide as that of benzoyl gave with alkali more than 88 per cent. of its contained sulphur in the form of thiocyanic

acid, and, what is still more remarkable, when treated with alcoholic ammonia yielded more than 50 per cent. of its sulphur as ammonium thiocyanate (Dixon and Taylor, *loc. cit.*). Amongst purely hydrocarbon thiocarbimides, this elimination of the rhodanic group is not known to occur; in fact, it does not usually happen even with hydrocarbon thiocyanates, which tend to yield mercaptans and the like.

Again, transformation of the $\cdot\text{SCN}$ group into $\cdot\text{NCS}$ is by no means uncommon, even when the associated radicle is not definitely electronegative, as in the case of allyl thiocyanate. If conjoined with a distinctly acid radicle, and hence but loosely held, the rhodanic group could still more readily alter its mode of attachment, and would presumably do so in whatever manner leads to the most stable kind of union between the two constituent groups. As to electrical character, it is not very easy to compare the two configurations of the rhodanic complex. But seeing that the thiocyanates of the alkali metals are neutral saline compounds, the residue $\cdot\text{SCN}$ must be strongly electronegative, and in this respect comparable with the residues of the mineral acids. On the other hand, the known combinations of the group $\cdot\text{NCS}$ do not appear to be saline, nor does it unite with the metals of the alkalis or alkaline earths; on these grounds, at least, the group $\cdot\text{NCS}$ may be considered less electronegative than the group $\cdot\text{SCN}$. Consequently, if a strongly acid radicle is conjoined, even for the moment, with the group $\cdot\text{SCN}$, the latter might pass rapidly into the more electropositive configuration $\cdot\text{NCS}$. On similar principles, the rhodanic group if transferred to a distinctly electropositive radicle, such as K, Na, etc., may be compelled to assume the form, $\cdot\text{SCN}$. This would seem to be the case, for when acetylthiocarbimide is treated with sodium ethoxide in presence of benzene, the action takes place as follows:



(Dixon, *Trans.*, 1904, 85, 353). The rhodanic group of acetylthiocarbimide, which is desulphurisable by alkaline salts of lead or silver, is transferred, not to the ethyl, but to the sodium, and when there attached, has lost the property of becoming desulphurised; had it passed (as $\cdot\text{NCS}$) to the ethyl group, the new rhodanide would have been desulphurisable; presumably, therefore, the configuration of the group is changed.

Since alkalis and alkaline nitrogenous bases tend to hydrolyse the acyl thiocarbimides with production of thiocyanic acid, whilst non-alkaline bases, such as the primary arylamines, have much less effect in this way, it might be anticipated that still more feeble bases—

such, for instance, as diphenylamine—would show even less power to cause hydrolysis, and, concurrently, more to enter into thiocarbimide union. Adequate material on which to judge is still wanting, but so far as may be learned from the behaviour of acetylthiocarbimide, this appears to be the case (compare Doran, *Trans.*, 1905, **87**, 341; Dixon and Hawthorne, *loc. cit.*; Dixon and Taylor, *loc. cit.*). It seems, therefore, not improbable that what conditions the "thiocyanic" decomposition of acyl thiocarbimides is not any reluctance on the part of the $\cdot\text{NCS}$ groups to enter into ordinary thiocarbimide reactions, but rather the tendency of the electropositive material offered for combination to seize instead directly on the electronegative group of the thiocarbimide, and so to liberate its rhodanic radicle.

If it be true that strong electronegative character in an organic radicle may entail the attachment of the rhodanic group solely as $\cdot\text{NCS}$, it is reasonable to anticipate the existence of a similar mode of union amongst the rhodanides (at present known as "thiocyanates") of well-marked non-metallic radicles.

Hitherto but few non-metallic rhodanides have been prepared, the only certainly known examples being the thiocyanates of phosphorus, silicon, and arsenic (Miquel, *loc. cit.*), together with phosphoryl "thiocyanate" (Dixon, *Trans.*, 1901, **79**, 541). The silicon derivative has been re-examined carefully by Emerson Reynolds, who concludes (*Trans.*, 1906, **89**, 204) that it is really a thiocyanate; on the other hand, the phosphorus and phosphoryl compounds have been studied to some extent from the chemical point of view by one of the recent writers (Dixon, *loc. cit.*), with somewhat different results.

The latter study has now been resumed, in the hope of learning by physical methods something further as to the constitution of these two rhodanides. To this end, the molecular refractions have been measured, but, since the inferences drawn rest on the interpretation of the numerical results, it seems desirable to indicate by reference to other cases how the experimental figures have been applied towards the solution of the problem.

Using the molecular refraction M_D obtained from the formula:

$$M_D = \frac{p(\mu_D - 1)}{d_{40}^{\circ}},$$

where p = molecular weight, μ_D = index of refraction for the spectral line, D , and d_{40}° = density at 40° referred to water at 4° , Hawthorne found (*loc. cit.*) for methyl and ethyl thiocyanates at the ordinary temperature the figures 32.1 and 40.2 respectively. These, less 9.0 and

16.7—the refraction values for CH_3 and C_7H_5^* —gave as the effect of $\cdot\text{SCN}$, 23.1 and 23.5, or a mean of 23.3 units.

Ethylthiocarbimide and allylthiocarbimide gave at 15° , after deduction of the calculated values for the hydrocarbon radicles, 28.0 and 27.63 for $\cdot\text{NCS}$, and benzylthiocarbimide, after deduction of the mean value observed for C_7H_7 (obtained from chlorotoluene and from benzyl chloride), gave 27.84, or a mean refraction effect for $\cdot\text{NCS}$ of 27.82. When this method was applied to acetyl rhodanide (M_D at $20^\circ = 46.1$), the value 17.8 for acetyl being deducted, the difference, 28.3, lay so near to the above figure for $\cdot\text{NCS}$ that he decided in favour of the constitution represented by the formula $\text{CH}_3\cdot\text{CO}\cdot\text{NCS}$.

The mean of all these values for $\cdot\text{NCS}$ is 27.92, which differs by roughly one-third of a unit from the highest and lowest obtained.

Cyclic thiocarbimides, for some reason not yet understood, give results far in excess of the calculated numbers. It is possible, nevertheless, by determining the effect of the cyclic group in conjunction with CNS in a known compound, and by allowing for the former, to ascertain the refraction value of the rhodanic group when conjoined with similar benzenoid radicles. Thus, if from the mean number for phenylthiocarbimide, namely, about 77.6 (Nasini and Scala, *Gazzetta* 1886, 16, 70; Berliner, *Diss.*, Breslau, 1886), there be deducted what the present writers consider the most probable value for $\cdot\text{NCS}$, namely 28.1, the difference, 49.5, represents approximately the refraction value of the phenyl group in such combinations. For example, it was found (Dixon and Taylor, *Trans.*, 1908, 93, 692) that for *o*-tolylthiocarbimide, $M_D = 85.8$; this, less 49.5 (Ph) and 7.7 (CH_3), gives 28.6, a number not far from 28.1, the above "most probable value" for $\cdot\text{NCS}$.

Benzoyl rhodanide gave (*loc cit.*) $M_D = 85.7$, which, less 49.5 and 8.5 (C_6H_5 and $\text{C}\cdot\text{O}$), leaves 27.7; from this it was concluded that the substance is a thiocarbimide. In like manner, carboxymethyl and carboxyethyl rhodanides, $\text{CO}_2\text{Me}\cdot(\text{CNS})$ and $\text{CO}_2\text{Et}\cdot(\text{CNS})$, when compared with acetylthiocarbimide on the lines indicated, gave for the $\cdot\text{NCS}$ group the figures 28.7 and 27.9 respectively; of the several values given above for $\cdot\text{NCS}$, the mean is 28.1, a number differing approximately by one-half unit from the highest and the lowest. Considering the difficulty of obtaining and of preserving these substances in a pure condition, that in calculating the molecular refraction the specific refraction ($\mu_D - 1/d$), and hence the experimental error in its determination, may have to be multiplied by very large number (over 160 in the case of benzoylthiocarbimide), and lastly, that the end results are but differences depending ultimately on the precision

* The atomic refractions used were:

H = 1.306; C = 5.092; C=C = 12.25; O = 3.426; Cl = 9.864.

with which the other factors are measured and on a constant value being obtained by these different combinations, the concordance of the various refraction values for the thiocarbimide group seems reasonably close.

Whilst, then, the results are obviously no more than mere approximations to the exact figures (if, indeed, there be definite values at all), they serve, nevertheless, to show that the group $\cdot\text{NCS}$ exerts on light a tolerably constant effect, which is measurably different from that produced by the isomeric configuration $\cdot\text{SCN}$. To ascertain whether the refraction number, about 23.3, already obtained for the latter holds in the case of other than purely aliphatic hydrocarbon radicles, we have now examined benzyl thiocyanate, the molecular refraction in alcoholic solution being calculated from the formula :

$$100 \frac{(\mu - 1)}{d} = \frac{(\mu' - 1)}{d'} r + \frac{(\mu'' - 1)}{d''} (100 - r),$$

where μ , μ' , and μ'' = indices of mixture, substance, and solvent respectively for the D line, and d , d' , and d'' = densities corresponding, r being the weight of substance in 100 parts by weight of the mixture.

The mean of two determinations in solutions containing respectively about 11 and 13 per cent. of thiocyanate was 75.7. This, less 52.3 for the benzyl radicle (see above), leaves for the $\cdot\text{SCN}$, 23.4, a figure agreeing closely with the mean value obtained in other cases, namely, 23.3.

On the basis, therefore, that a refraction number for the rhodanic group of about 23.3 units on the above scale implies the configuration $\cdot\text{SCN}$, and that of about 28.1 units the configuration $\cdot\text{NCS}$, we have examined the supposed thiocyanates, $\text{P}(\text{SCN})_3$ and $\text{PO}(\text{SCN})_3$.

Phosphorus Trirhodanide, $\text{P}(\text{CNS})_3$.

The preparation of this compound having already been described (Dixon, *loc. cit.*), details of the procedure are unnecessary. Instead, however, of ammonium thiocyanate, the hygroscopic character of which entails some difficulties, the corresponding lead salt was employed. It may be noted, also, that the bumping and frothing during distillation, which are always more or less troublesome, are considerably lessened if the phosphorus trichloride is freshly distilled before use.

The boiling points previously recorded were confirmed, a specimen twice rectified distilling at $164\text{--}165^\circ$ under a pressure of 16 mm. ; the density, given before as 1.487 at $15^\circ/4^\circ$ (different specimens when washed with water and redistilled gave also 1.483 and 1.488), was now found at $15^\circ/4^\circ$ to be 1.483.

The refractive index of the oil approaching very nearly to the limit

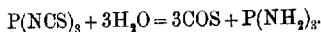
of the highest prism in the Pulfrich refractometer employed, its value was determined by the method of mixture specified above. In this way, μ_D was found to be 1.71739 at 15° ; whence $M_D = 99.17$.

The refractive index of phosphorus trichloride, as determined by Gladstone and Dale (*Phil. Trans.*, 1863, 153, 341), when combined with the careful determinations of its density by Thorpe (*Trans.*, 1880, 37, 334), gives for M_D at 28.5° and at 38° , 44.96 and 44.97 respectively. This we checked, finding for μ_D at 150° the figure 1.5152; whence $M_D = 44.7$. A determination by Nasini and Costa (*Pub. dell. Ist. chim. Roma*, 111, 1891) gives by interpolation for the *D*-line the value 45.2; the mean of these three is 44.95. The refraction of the three chlorine atoms being in round numbers 3×9.9 , or 29.7, the difference for the phosphorus is 15.25.

Deducting now from the molecular refraction of phosphorus tri-rhodanide the above value for phosphorus, we have for the joint total effect of the three rhodanic groups $99.17 - 15.25 = 83.92$, or, assuming them all to be equal, about 28 units for each.

So far, therefore, as this purely physical method of examination may be relied upon as affording evidence of chemical structure, the conclusion seems justified that phosphorus tri-rhodanide is *not* a thiocyanate (M_D for which would be about 85.1 units, instead of the 99.2 found), *but* a thiocarbimide.

Chemical evidence to the same effect, as might be anticipated, is more difficult to secure. For, as it has been mentioned already, the rhodanides of negative organic radicles invariably may be hydrolysed with formation of thiocyanic acid; and it is doubtless by reason of the ease with which in many cases this change takes place that such particular compounds as show the reaction have generally been accepted as thiocyanates. But, since the mere production of thiocyanic acid by hydrolysis is plainly insufficient to characterise a given organic acid rhodanide as a thiocyanate, the formation of this substance in the case of an electronegative inorganic rhodanide can equally afford no clue as to the constitution of the material which yields it. Indeed, considering the high affinity of phosphorus for oxygen, and its relatively slight tendency to form amino-compounds, one could scarcely anticipate the occurrence of the "thiocarbimide" hydrolysis:



Only by cautious treatment, therefore, avoiding the presence of water or strong bases, might one reasonably expect to make manifest the thiocarbimide power of a compound the molecule of which is obviously prone to total disruption (Miquel, *loc. cit.*). To present in the absence of water a feeble base seemed most hopeful, since the hydrolytic effect (or the tendency of the basic residue to withdraw

the phosphorus) would be minimised, thereby giving to the supposed $\cdot\text{NCS}$ groups a chance to enter into their characteristic thiocarbamidic combinations.

To secure these conditions, aniline and the still more feeble diphenylamine were employed; so far successfully, that definitive, additive thiocarbamidic combination has been accomplished, although not to the full extent betokened by the presence of three $\cdot\text{NCS}$ groups. But that additive change may to some extent be realised is shown by the following experiments.

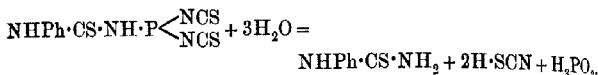
Phosphorus Trithiocarbimide and Aniline.—To 4.93 grams of freshly-distilled oil, dissolved in cold benzene, 2.22 grams of pure aniline, also in benzene, were added (instead of 2.24, the calculated weight for equal molecules). The precipitated granular solid was filtered off, and to the filtrate were added two or three drops more of aniline, this causing a slight further precipitate; on concentrating somewhat and filtering again, the filtrate when treated with aniline now remained clear. The precipitates, after drying, weighed in all 7.15 grams; the amount calculated for an equimolecular compound being 7.176 grams, that obtained represents 99.6 per cent. of the theoretical.

In the main product, a cream-white powder, ready-formed aniline thiocyanate was absent, for when the solid was shaken up with water the aqueous portion gave with ferric chloride a barely perceptible colour change, and no detectable reaction for aniline. On standing with cold water, hydrolysis occurred slowly, the reaction with hydrochloric acid and ferric chloride becoming gradually more marked, and, after some four or five days, intense. On boiling, the thiocyanic reaction was obtained at once, a perceptible trace of hydrogen sulphide being evolved; aniline was now detectable, although in very small amount, and phenylthiourea crystallised out as the solution cooled.

That the phenylthiourea did not result through ordinary isomeric change of aniline thiocyanate, conceivably formed in the hydrolysis, was established by the following observations. Aniline thiocyanate, when heated to boiling in dilute aqueous solution together with a little alcohol, yielded in one minute no perceptible amount of phenylthiourea, as tested by the action of boiling alkaline lead or silver salts on the solution; after three or four minutes' boiling, a very slight desulphurisation began to show, which increased steadily as the time of boiling of the aqueous solution was prolonged; in the cold, no reaction was noticeable after many hours' standing. On the other hand, the solution of the phosphorus compound in alcohol gave with silver nitrate a white precipitate, which was blackened gradually on standing in the cold, or instantly on warming. The compound gave also with cold dilute alkali an opalescent solution; a portion of this,

when acidified, gave with ferric chloride an intense red coloration; another portion, when treated with an alkaline lead salt, gave a primrose-yellow precipitate, which became black, gradually in the cold, or immediately on gentle warming. Phenylthiourea, dissolved in alkali hydroxide, behaved so far as the desulphurisation with lead solution is concerned in precisely the same way, save that the darkening was somewhat slower in the cold.

It has already been shown (Trans., 1904, 85, 359) that of the total sulphur contained in this compound, about 4 per cent. escapes on rapid hydrolysis as hydrogen sulphide, and that of the remainder, about one-third crystallises out as phenylthiourea (in practice, nine-tenths of this fraction was collected), whilst about two-thirds appear as thiocyanic acid. Apart, therefore, from the slight change first mentioned, the main decomposition may be represented thus:



Very remarkable is this sharp union in the cold of a single molecule of aniline with but one of the three $\cdot\text{NCS}$ groups in the tri-thiocarbimide. Possibly, but not very probably, it may be due to steric hindrance that the remaining groups fail to combine; in this case, however, one would rather have anticipated the ready union of two $\cdot\text{NCS}$ groups with aniline, the difficulty arising in connexion with the third. Cases, however, are not uncommon in which, if several like atoms or atomic complexes are charged upon a single other atom, the atoms or complexes so conjoined show inequality of power to interact. Thus, for example, of the two chlorine atoms in carbonyl dichloride, but one reacts very readily with alcohol, the resultant ethyl chlorocarbonate uniting only slowly with excess of alcohol. Carbonyldithiocarbimide, $\text{CO}(\text{NCS})_2$, too, when mixed with alcohol, takes up but one molecule to form $\text{SCN}\cdot\text{CO}\cdot\text{NH}\cdot\text{CS}\cdot\text{OEt}$ (Dixon, Trans., 1903, 83, 87). With bases, it combines readily in equimolecular proportions; two molecules of base are not taken up in any case so readily as one, and in certain instances (namely, with naphthylamine and with benzyaniline), even although excess of base is used, a single molecule alone enters into combination. Similar instances might be multiplied, but it would serve no other purpose than to show that our knowledge of the chemical power of a radicle, singly held, cannot always be extended by mere arithmetic to cases where there are two or more, especially if these be attached to one particular atom.

Phosphoryl Trirhodanide, PO(CNS)₃.

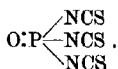
In preparing this substance, potassium and lead thiocyanates were tried, and, as solvents in each case, benzene or cumene, the best yield being obtained from the lead salt with cumene. Previous distillation of the phosphoryl chloride was found very advantageous in reducing the tendency of the solution to bump and froth during the fractionation; commercial cumene, which contains naphthalene, is unsuitable, for the latter, unless removed, is left ultimately mixed with the rhodanide, and causes difficulty.

The solvent was distilled off under diminished pressure by the aid of a water-bath; the residual oil was then collected separately and rectified. Its properties agreed with those already described (*loc. cit.*), the boiling points observed in various preparations being 159°/11 mm., 160°/12 mm., and 164°/14 mm., the previous figure being 175°/21 mm. Its density, the mean of three closely concordant determinations from different specimens, was 1·518 at 15°/4°. The refractive index at about 15° was found to be, as the mean of three concordant determinations for material once rectified in each case, $\mu_D = 1·6882$, and from three preparations, each doubly rectified, $\mu_D = 1·6918$. From these figures, $M_D = 100·02$ and $100·48$ respectively; the latter value is used below.

Since the phosphoryl group, as shown by Gladstone (Trans., 1870, 23, 112), has an abnormally low refraction effect, at least in meta- and ortho-phosphoric acids, it was necessary to ascertain directly what the value is in phosphoryl chloride, POCl₃. From Gladstone and Dales' determination (*loc. cit.*), namely, $\mu_D = 1·4882$ at 17°, and Thorpe's careful measurement of its density (*loc. cit.*), namely, 1·6805 at 17°/4°, M_D for POCl₃ = 44·6. This, less Cl₃ (3 × 9·9, or 29·7), leaves for the group :PO the value 14·9, something less than that of the phosphorus which it contains.

Consequently, the total effect of the rhodanic groups in phosphoryl rhodanide is about 100·5 – 14·9, or 85·6, one-third of which (assuming them to be alike), or 28·5, gives the value of each; this figure is obviously much nearer to that representing ·NCS (28·1) than the 23·3 which measures the effect of ·SCN. If phosphoryl rhodanide contained three of the latter groups at the value named, M_D would be 84·8, instead of about 100·5.

Judging from the above results, the conclusion appears to be justified that *phosphoryl rhodanide*, like the corresponding phosphorus compound, is not a thiocyanate, but a thiocarbimide,

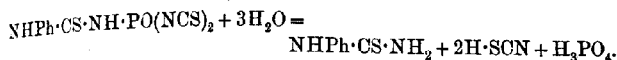


Notwithstanding that phosphoryl trithiocarbimide is hydrolysed with ease into phosphoric and thiocyanic acids, there is no difficulty in obtaining also from it the reactions characteristic of a thiocarbimide. For example, the cold alcoholic solution, when treated with alkaline lead tartrate, gave a turbid mixture, which very quickly became black, owing to desulphurisation. Silver nitrate, followed by ammonia in the cold, gave a like result, and mercuric or cadmium salts in presence of alkali yielded at once, on gentle warming, the corresponding black or yellow sulphide. To ascertain to what extent the thiocyanic hydrolysis is accomplished in presence of alkali and water, a weighed quantity of the oil (about $1\frac{1}{2}$ grams) was dissolved in hot water containing 5 equivalents of *N*-sodium hydroxide; the faintly alkaline solution was neutralised exactly by very dilute nitric acid, made up to a known volume, and boiled to expel a trace of hydrogen sulphide; the contained thiocyanic acid was then determined by Barnes and Liddles' method, using *N*/10-copper sulphate. Of the total sulphur present in the oil, 90.75 per cent. was found as $\text{H}\cdot\text{SCN}$, a figure close to that obtained for benzoylthiocarbimide and for carboxyethylthiocarbimide, namely, about 89 and 93 per cent. respectively (Dixon and Taylor, *loc. cit.*).

Phosphoryl Trithiocarbimide and Aniline.—To a benzene solution of the freshly-distilled oil, one molecular proportion of aniline was slowly added, the latter also being dissolved in benzene; the weight of the yellow, granular precipitate which formed at once, together with that of another small quantity which separated from the mother liquor on standing, amounted to precisely the sum of the weights of materials employed. The melting point, 121.5° , with previous softening at 120 – 121° , was practically identical with that observed before (Dixon, *loc. cit.*), and a sulphur determination, made as a check, fell a fraction of a per cent. below the figure calculated for an equimolecular compound. Cold water, shaken up with the solid, extracted from it scarcely a trace of thiocyanic acid, and no detectable amount of aniline; on standing, however, hydrolysis began to occur, the process running a course exactly similar to that with the corresponding phosphorus derivative. The action is very slow at the ordinary temperature; thus, in an experiment where a quantity of the additive compound was mixed with about twenty times its weight of water, the mixture being shaken up frequently during a fortnight, thiocyanic acid and phosphoric acid passed into the solution in slowly increasing amount; but on washing the residual solid until the filtrate ceased to react for thiocyanic acid when tested with ferric chloride, and again leaving the solid residue in contact with water, these two acids continued as before to accumulate in the solution.

With dilute alkali, or with hot water, the hydrolysis takes place

rapidly. Thus, when a weighed quantity of the solid was digested on the water-bath for twenty minutes with a slight excess over four equivalents of $N/10$ -alkali, together with some water, and the solution just acidified and diluted to a known bulk, the latter mixture, by comparison with a standard solution of thiocyanate and ferric chloride, was found to contain sixteen-seventeenths of the thiocyanic acid, which could result from the change



The colorimetric method, according to our experience, working at a concentration of $N/1000$, gives results which are accurate to within about 5 per cent. of the total amount evaluated. Barnes and Liddle's process proved unavailable, the phenylthiourea formed in the hydrolysis combining with the copper solution to produce some double compound, the nature of which we did not investigate beyond verifying that it is also precipitated when copper sulphate is added to phenylthiourea in presence of alkali bisulphite; it is not formed from these two in the absence of the bisulphite.

These results are so far confirmatory of those previously obtained (*loc. cit.*) as to establish with reasonable certainty the propositions: (i) that phosphoryl trirhodanide in the cold enters at once into definite chemical combination with a single molecule of aniline, one only of its three rhodanic groups thereby becoming engaged with the base added; (ii) that, apart from a trifling side-reaction leading to the production of hydrogen sulphide, the additive compound is hydrolysed very readily into phosphoric acid, two molecules of thiocyanic acid, and one molecule of phenylthiourea; (iii) that the original additive compound does not contain aniline thiocyanate, neither does the phenylthiourea produced by the hydrolysis result from the transformation of aniline thiocyanate, conceivably formed during the act of hydrolysis.

What occurs, then, in the combination of aniline in the circumstances named is this: of the three rhodanic groups attached to the phosphoryl residue, one alone absorbs the base, entering with it into definite thiocarbamidic combination; the other two remain intact. On hydrolysis, the phenylthiocarbamido-complex is removed as phenylthiocarbamide (or phenylthiourea); the hydrolysis is completed by the elimination of the two remaining rhodanic groups, during which, as with other acylthiocarbimides, a small fraction of the sulphur escapes by thiocarbimide hydrolysis as carbonyl sulphide, the main part of it decomposing as (CNS) and yielding thiocyanic acid.

The next point to be decided was whether more than one molecule

of aniline can be united with a molecule of the trirhodanide. Since a single molecular proportion of aniline precipitates and removes completely from benzene solution the whole of the dissolved rhodanide, the experiment was now reversed, one molecular proportion of the phosphorus compound being added, drop by drop, in cold benzene to two molecular proportions of aniline, similarly dissolved, and kept cool. As before, a precipitate formed; when washed with benzene and dried in the steam-bath, its weight amounted to 99.8 per cent. of the total weight of material employed for combination. The benzene mother liquor contained a mere trace of thiocyanic acid, but no detectable amount of aniline.

The product, faint yellowish-white in colour, shrank at 113° and melted between 114.5 and 115.5° . On analysis:

0.407, fused with $\text{NaOH} + \text{KNO}_3$, gave 0.600 BaSO_4 . $S = 23.2$.

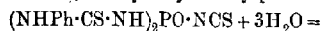
$\text{C}_{15}\text{H}_{14}\text{ON}_3\text{SP}$ requires $S = 23.58$ per cent.

The compound therefore is $\text{PO}(\text{NCS})_3 + 2\text{C}_6\text{H}_5\cdot\text{NH}_2$.

As in the case of the preceding compound, aniline thiocyanate was not present ready formed, for the solid when shaken up with water was practically insoluble, the aqueous portion giving with hydrochloric acid and ferric chloride a barely perceptible red coloration, but, on boiling, the reaction was given abundantly.

Strange to say, the second molecule of aniline is not held in the same thiocarbamidic combination as the first, or if it is, the decomposition occurs differently. For when the compound was hydrolysed as in the preceding case, and the liberated thiocyanic acid measured colorimetrically (Barnes and Liddles' method proving inapplicable), the amount of this acid formed, as nearly as can be judged by this process, was that corresponding with two-thirds of the total sulphur present. The hydrolysed mixture gave the reaction for aniline.

How to explain this curious behaviour, save conjecturally, we do not yet know, but on the ground that aniline thiocyanate is not present as such in the product (which, moreover, was at no stage of its formation pasty, as is generally observed if this substance is formed), the hydrolysis may provisionally be represented as follows:



When one molecular proportion of the rhodanide was added to three molecular proportions of aniline in the cold, a solid separated, but part of the base did not enter into combination, for the benzene liquor reacted freely for aniline. On warming the mixture on the water-bath to about 65° , the solid melted to a clear oil, and the benzene now no longer gave the reaction for aniline. Apparently, therefore, three molecules of the base had been absorbed, but on

running off the mother liquor, and allowing the oily residue to stand, it slowly changed into a yellow, pitchy resin, so unlike a definite chemical compound that no attempt was made to analyse it. With cold water the powdered resin behaved somewhat like the preceding compounds, gave that the mixture when acidified and treated with ferric chloride gave a distinct, although not strong, red coloration.

Phosphoryl Trithiocarbimide and Diphenylamine.—From 10.574 grams of thiocarbimide and 7.14 of diphenylamine (equal molecules), both in benzene, there separated 17.5 grams of a faintly yellow product consisting of microscopic needles. When heated in a narrow tube, these shrank slightly at 138°, and melted between 140° and 141° (uncorr.). A sulphur determination showed the substance to be an equimolecular compound:

0.377, fused with NaOH + KNO₃, yielded 0.662 BaSO₄. S = 24.2.

C₂₇H₂₀ON₅S₃P requires S = 24.6 per cent.

Cold water had little effect on this diphenyl compound, the mixture at the end of four days, during which it was frequently shaken up, giving with ferric chloride no more than a faint red coloration; on boiling, a well-marked reaction was obtained.

Under the action of dilute alkali hydroxide, hydrolysis occurred readily; thus, when 5.85 grams of the solid were suspended in hot water, and *N*-sodium hydroxide added, drop by drop, until the solution became just alkaline to litmus, 48 c.c. were required. Since, for the above quantity of substance, each 15 c.c. of normal solution represents one equivalent, a little more than three equivalents had been absorbed. When cold, the mixture was separated by filtration from the white solid, which now no longer yielded thiocyanic acid when boiled with potassium hydroxide, and was free from phosphorus; it was recrystallised from alcohol, and identified as *aa*-diphenylthiocarbimide. Its weight, apart from what remained in the aqueous portion (for the substance is not quite insoluble in water), amounted to 3.08 grams, that is, nine-tenths of what could be formed according to the equation:



All the phosphorus had passed into the aqueous filtrate, which reacted intensely for thiocyanic acid, and gave the usual reactions for phosphoric acid; it contained, in addition, a trace of dissolved thiocarbimide.

Phosphoryl trithiocarbimide, when added to two molecular proportions of diphenylamine dissolved in cold benzene, yielded the above unimolecular additive compound in a somewhat impure condition; the mother liquor, when concentrated by evaporation, afforded a further small crop of a mixture melting between 150° and 165°, and, when

allowed to evaporate to dryness, left a residue containing much unchanged diphenylamine.

From these results it would appear that diphenylamine, in the circumstances given, is less disposed than aniline to form with phosphoryl trithiocarbimide a compound containing two molecules of the base. In this connexion, it may be recalled that, whilst the dithiocarbimide of carbonic acid, $\text{CO}(\text{NCS})_2$, can unite with either one or two molecules of aniline or of toluidine, it combines with but a single molecule of naphthylamine or of benzylaniline, even although the latter bases be present in excess (see p. 2156).

Earlier in this paper it has been suggested that the mode of union of a rhodanic group may depend on the character of the radicle to which it is attached. Since hydrogen, like the hydrocarbon residues, represents a kind of electrical mean between strongly electropositive radicles, such as K, Na, etc., and strongly electronegative, such as CH_3CO , $\text{O}\cdot\text{P}$, and the like, it also might be expected to yield the two rhodanides, $\text{H}\cdot\text{SCN}$ and $\text{H}\cdot\text{NCS}$.

It seems probable, however, that the great mobility of hydrogen, coupled with its generally electropositive character, may suffice to preclude its permanent association with the rhodanic complex, save in the one form $\text{H}\cdot\text{SCN}$, and similarly with other radicles, such as CNO , CN , NO_2 , etc., which with hydrocarbon residues can give isomeric combinations, whereas with hydrogen they yield in each case but a single form. However this may be, the fact remains that when a thiocarbimide is hydrolysed, the products may contain carbonyl sulphide or thiocyanic acid, but not hydrogen thiocarbimide, $\text{H}\cdot\text{NCS}$.

It being tolerably clear, in a general way, that the less electropositive the base presented for interaction to an acidic thiocarbimide the less is the resulting thiocyanic decomposition (and concurrently, the greater is the percentage of thiocarbamidic union), the idea occurred of trying whether chemical action could be brought about with distinctly electronegative "bases." For, although the amides do not combine directly with thiocarbimides containing hydrocarbon radicles, yet diphenylamine, which is little disposed to unite with these, does so vigorously with many acylthiocarbimides, and hence it seemed worth while to experiment on the lines indicated.

The combinations attempted were: (i) benzoylthiocarbimide in benzene or acetone with oxamide, benzamide, acetanilide, and urethane, all in the cold; (ii) carboxymethylthiocarbimide with benzamide and with urethane, also in the cold; (iii) acetylthiocarbimide with urethane in boiling toluene. In no case could any sign of combination be detected.

It is plain, therefore, that a certain amount of "basicity" is requisite for the combination of "base" and thiocarbimide; more-

ver, acetylthiocarbimide can furnish with aniline the compound *N*-acetylphenylthiocarbimide, whilst phenylthiocarbimide, it appears, cannot unite with acetamide to yield the same compound. Yet the hydrogen of the NH_2 -group in acetamide is not especially difficult of removal, for it may be replaced directly either by bromine or by sodium. Ethylthiocarbimide, too, fails to combine with sodium acetanilide (Dixon, *Trans.*, 1899, **75**, 384), and phenylthiocarbimide with asparagine (*ibid.*, 410); neither does acetyl chloride react with phenylthiocarbimide dissolved in aqueous sodium hydroxide (Dixon and Hawthorne, *loc. cit.*), a liquid supposed to contain the sodium derivative of phenylthiourea. In the present state of our knowledge, to explain these facts is more than difficult.

Although the only organic acyl rhodanides so far isolated are those derived from carboxylic acids, there is some reason to hope that certain non-carboxylic acids may afford similar products. For when the chlorides of phenylsulphonic and ethylsulphuric acids respectively were heated in presence of hydrocarbon solvents with lead thiocyanate, the liquor in each case was desulphurised by lead and silver salts, and hence may have contained dissolved thiocarbimide (Dixon, *Trans.*, 1897, **71**, 640). Owing to the difficulty of securing complete interaction without decomposing the organic product, these experiments were abandoned until better methods should become available; it is now intended to resume the study, in the hope that more satisfactory conditions may be realised. It is proposed, also, to examine the molecular refraction of rhodanides containing inorganic radicles other than those of phosphorus and phosphoryl, with the view of learning whether, in such cases, the above physical method will serve as a means of distinguishing statically between the thiocyanic and the thiocarbimidic form of linking.

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CCXVI.—*Ester Catalysis and a Modification of the Theory of Acids.*

By EDWARD FITZGERALD and ARTHUR LAPWORTH.

THE main facts relating to the phenomena of catalysis by acids are well known. They have been studied in the main with reference to esterification and ester and sucrose hydrolyses. The influence of acids as catalysts is known to be exerted in many other types of reaction, as,

for example, in the bromination of carbonyl compounds, the decomposition of diazoacetic ester (Bredig and Fraenkel, *Ber.*, 1906, 39, 1756), and the transformation of hydrazobenzene into benzidine. The numerous theories which have been devised to explain the catalytic influence of acids on esterification and ester hydrolysis have recently been dealt with in a fairly complete manner by Acree (*Amer. Chem. J.*, 1908, 39, 145), and therefore it is not necessary fully to review the question here from the historical standpoint. It is sufficient to say that Kastle appears to have been the first (*Amer. Chem. J.*, 1897, 19, 894) to have suggested the view now most generally held, namely, that the ions of the acid acting catalytically react with the substance undergoing change, forming an unstable addition product which subsequently yields the final products; it is evident, however, that the same natural idea has occurred independently to other chemists. The view now most generally held in reference to ester catalysis, is that the carbonyl compound forms a complex with the hydrogen ions. On the other hand, Goldschmidt and Udby (*Zeitsch. physikal. Chem.*, 1907, 60, 728) claim to have established that it is the alcohol which mainly forms the reacting initial addition compound or complex hydrion, but this claim cannot be accepted.

The facts on which a recent discussion of the mechanism of esterification and ester hydrolysis have been based (Acree and Johnson, *Amer. Chem. J.*, 1907, 38, 301) are: that the velocity of catalysed esterification and ester hydrolysis are usually nearly proportional to the concentration of catalyst, where the latter is a powerful acid, and to the concentration of the carbonyl compound (ester or carboxylic acid); and further, that the activity of the catalyst is roughly proportional to its so-called degree of dissociation as given by conductivity measurements.

A slight lowering of the conductivity of acids was found by these authors to be produced by the addition of carboxylic ester. But their subsequent discussion of the question is based on the assumption that the velocity of esterification and ester hydrolysis is intrinsically proportional to the concentration of the alcohol and water respectively. This assumption is one which is made in most text-books on Physical Chemistry, but was not sufficiently well justified to warrant the rejection of any theory of esterification which did not fulfil this condition.

It must be pointed out that the classical experiments of Berthelot and Péan de Saint-Gilles do not throw any light on the question. Further the condition at equilibrium in a mixture of alcohol, carboxylic acid, ester, and water is determined by thermodynamic laws and must be the same whatever the path by which it is attained. The mechanism can only be decided after a study of the conditions

affecting the initial rate of change in the absence of the products of change.

A theory put forward by one of us having been assailed on the above ground, experiments were made showing that, in point of fact, the initial velocity of hydrolysis of an ester in acetone is nearly independent of the water concentration over a wide range (Proc., 1908, 24, 101, 152), but the erroneous conclusion was drawn that, on this account, theories demanding a proportionality between the velocity and water concentration were wrong. The equivocal character of the results was clear from the fact that, within the same range of water concentration in acetone, the initial velocity of catalytic esterification is roughly inversely proportional to the concentration of the water, but proportional to the concentration of the alcohol, which is its analogue in the reverse reaction. The numbers obtained by Kistiakowsky (*Zeitsch. physikal. Chem.*, 1898, 27, 253 *et seq.*) also show that, within similar limits, similar relations obtain, whilst Goldschmidt (*Ber.*, 1896, 29, 2308 *et seq.*) was the first to draw attention to the very great depression produced by small quantities of water on the velocity of esterification in alcoholic solution.

In addition to the points above referred to, it was found by Fitzgerald and Lapworth (*loc. cit.*) that alcohol produced a very slight lowering only on the velocity of catalytic hydrolysis in acetone, and that the initial velocity of esterification is roughly proportional to the alcohol concentration.

The solution of the question whether the initial velocity of hydrolysis of esters by acids and water is proportional to, or independent of, the concentration of the water was evidently necessary for the final determination of the question of the mechanism of ester catalysis. During the interval since the publication of the preliminary notes, the authors have been engaged in experiments on the velocity of esterification of acetic acid under varying conditions, but have come to the conclusion that rapid progress towards the solution of the problem is to be made along different lines, and after a due consideration of the facts referred to above.

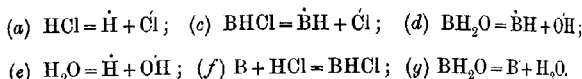
Their reasoning has been based on the belief that the alcohol and the water, so far as they take part in the esterification or ester hydrolysis, play similar parts. Now the velocity of esterification appears to be at the very first moment nearly proportional to the concentration of the alcohol, and therefore it seems impossible to avoid the conclusion that this is really also the case with the water, and that the general depressant effect of the increased concentration of water exactly neutralises the acceleration which would otherwise be observed. The most obvious view of the respective effects of alcohol

and water on ester catalysis is that, in solutions containing both, the two substances in question share between them some intermediate product, but on careful examination this fails to meet the case.

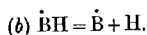
No consistent hypothesis can be advanced until the possibility of combination of the catalyst itself with the water is considered.

The theory advocated by the school of Arrhenius in the case of the hydrolysis of, say, aniline hydrochloride by water, is that, as the result of the dissociation of water in hydrogen and hydroxyl ions, the phenylammonium ions unite with hydroxyl ions from the water to form feebly dissociated phenylammonium hydroxide, and this, in turn, yields aniline and water. Further examination shows that this really explains hydrolysis by assuming the base to be converted into a relatively inactive hydrate. This is easily seen on considering the following expressions.

Let a small quantity of a weak base in a solvent containing hydrogen chloride and water be considered. The following equations represent the relations usually supposed to obtain, where B = free base, BH_2O the undissociated hydrate (ionisable + non-ionisable), $\dot{\text{B}}\text{H}$ the cation, BHCl the undissociated hydrochloride of the base. The equations are:



To these may be added a seventh, which is not so frequently introduced, but which is mathematically consequent on the others, namely,



Expressing concentrations by bracketing the symbols, the law of mass action leads to the following expressions, most of which are in general use:

$$\text{K}_1 = \frac{(\dot{\text{H}})(\dot{\text{Cl}})}{(\text{HCl})}, \quad \text{K}_3 = \frac{(\dot{\text{B}}\text{H})(\dot{\text{Cl}})}{(\text{BHCl})}, \quad \text{K}_4 = \frac{(\dot{\text{B}}\text{H})(\dot{\text{O}}\text{H})}{(\text{BH}_2\text{O})}, \quad \text{K}_5 = \frac{(\dot{\text{H}})(\dot{\text{O}}\text{H})}{\text{H}_2\text{O}}, \\ \text{K}_6 = \frac{(\text{BHCl})}{(\text{B})(\text{HCl})}, \quad \text{K}_7 = \frac{(\text{B})(\text{H}_2\text{O})}{(\text{BH}_2\text{O})}, \quad \text{and} \quad \text{K}_2 = \frac{(\text{B})(\dot{\text{H}})}{(\dot{\text{B}}\text{H})}.$$

Two of these being redundant, K_6 and K_7 may be conveniently expressed in terms of the others;

$$\text{hence} \quad \text{K}_6 = \frac{\text{K}_1}{\text{K}_2\text{K}_3} \quad \text{and} \quad \text{K}_7 = \frac{\text{K}_2\text{K}_4}{\text{K}_5}.$$

The following relations also result :

$$\begin{aligned}(\text{BH}_2\text{O}) &= \frac{1}{K_7}(\text{B})(\text{H}_2\text{O}) = \frac{K_5}{K_2 K_4}(\text{B})(\text{H}_2\text{O}), \\(\dot{\text{B}}\text{H}) &= K_4 \frac{(\text{BH}_2\text{O})}{(\text{OH})} = \frac{1}{K_2}(\text{B})(\dot{\text{H}}), \\(\text{BHCl}) &= \frac{1}{K_3}(\dot{\text{B}}\text{H})(\text{Cl}) = \frac{1}{K_2 K_3}(\text{B})(\dot{\text{H}})(\text{Cl}),\end{aligned}$$

which express the relationships between the concentrations of the compounds of B with the active masses of the hydrogen ions, the chlorine ions, and the water.

Whence it follows that if the hydrogen and chlorine ions remain nearly constant in amount or increase on addition of water,* the only way in which the salt and its cation can disappear is by conversion into undissociated BH_2O , as they are otherwise nearly independent of the amount of water present.

The application of the Arrhenius view, then, to the case of catalysis involves the assumption that the water combines with the substance affected, thus rendering it inactive. If so, when esterification in alcohol is considered, it must be the carboxylic acid which forms an inactive hydrate, as the alcohol is in enormous excess. This, apart from its inherent improbability, is not capable of explaining the depressions which are observed, and when the influence of water on the bromination of acetone, referred to in another paper, is considered, the explanation is totally inadequate and must be rejected.

The Hydrogen Ions of the Catalyst are Hydrated?—Inspecting the expressions above given for the equilibrium of a base in presence of an acid and water, it is seen that the only other explanation of the reduction in concentration of the salt or its cation is that the hydrogen ions disappear, that is, that water acts on them in virtue of its power of forming oxonium salts. The assumption that such complexes may be formed is in no way novel, and has been used by many chemists. What is novel in the present conception is that *the introduction of water into alcohol containing hydrogen chloride must necessarily reduce the number of hydrogen ions, or, in other words, of the amount of available hydrochloric acid.* Further, as acid catalysis, unless hydrolytic, occurs enormously faster in alcohol than in water, the number of hydrogen ions in an aqueous solution of hydrogen chloride must be extremely small, even compared with one in alcohol, which itself must be regarded as a base.

The mechanism of esterification in this view is intrinsically

* The increase in conductivity of hydrogen chloride dissolved in alcohol consequent on the replacement of alcohol by water is usually attributed to a greater degree of dissociation of the acids into H and Cl ions.

proportional to (alcohol) \times (carboxylic acid) \times (hydrion); that of hydrolysis to (water) \times (ester) \times (hydrion), and the apparent discrepancies between this, the usually accepted view, and experiment is due to alterations in the hydrion concentration which have not been foreseen. It is not yet possible to ascertain whether the alcohol and the water, on the one hand, or the ester and carboxylic acid, on the other, form the reactive complex ions, as the same results will be obtained in either case. The important work of Stieglitz on the hydrolysis of imino-ester ions and the properties of carbonyl compounds as a whole render the latter, and usual, view by far the more probable one.

This question, however, assumes a secondary importance as compared with the broader reflections to which its study has given rise. The proposition that free hydrogen ions are responsible for the catalytic activity of acids leads to the conclusion that they must be relatively few in number in aqueous solution. On the other hand, the original conception of hydrogen ions was applied to explain the conductivity of acids in aqueous solution, so that the terms are not synonymous.

In the latter case, they must be complex ions, probably of the form $\dot{\text{H}}_2\text{OH}$, if the analogy between water and ammonia is complete; the catalytically active hydrogen ions, if they have a real existence, are simpler than these, probably $\dot{\text{H}}$.

The view that both types exist will perhaps prove useful in the theoretical treatment of certain questions, but the alternative must be considered, for it has long been entertained in one form or another by a school of chemists, including Armstrong and Lowry.

The Velocity of Esterification when no Water is Present.

The velocity of esterification having been found to be over a wide range nearly inversely as the amount of water present, it was obvious that the form of the curve expressing the variation of velocity with the amount of water was roughly of the form of a rectangular hyperbola, but that the hyperbola would cut the velocity axis was tolerably certain, otherwise esterification with no water present would be instantaneous. This being highly improbable, it was clear that the curve near this point would have the form :

$$\frac{dx}{dt} \times [\text{H}_2\text{O} + b] = a,$$

when b and a were constants. The constant b was termed by the authors the water value of the solvent long before its true significance was understood.

The Theory of Goldschmidt and Udby.

Goldschmidt and Udby have discussed the question of the retarding effect of water on the catalytic esterification of carboxylic acids in alcohol (*Zeitsch. physikal. Chem.*, 1907, **60**, 728). These authors accept, without discussing the view of Arrhenius, the idea that the process of catalytic esterification depends on the intermediate formation of complex hydrions by one of the substances taking part in the change. They claim to prove that the alcohol must be the substance which mainly forms the reactive complex ions, but their process of reasoning would indicate that when the initial concentration of alcohol varied, the velocity would be unaltered, and this is contrary to experience.

They adopt the view that the complex water ions, H_3O , are the carriers of catalytic activity of acids in aqueous solution, and their views are thus essentially opposed to that advanced in the present papers, which is that these are usually, if not always, ineffective as catalysts.

Had these authors but perceived that the hydrogen ions, or the available hydrochloric acid, diminish in amount when water is added to alcoholic hydrogen chloride, they could not have formed the main conclusion which is put forward in their communication. Nevertheless, they must be credited with the first attempt to explain the anticatalytic effect of water on esterification in alcohol as the result of complex ion hydrolysis.

EXPERIMENTAL.

The experiments described were all carried out at $25 \pm 0.02^\circ$, using flasks which had been steamed and then dried by a current of hot air.

Except where a definite statement is made to the contrary, the total volume of solution was made up with pure acetone to a total volume of 25 c.c. The catalyst used throughout was hydrogen chloride, and during the reactions had the concentration 0.0123 *N*. It was found undesirable to prepare the preliminary solutions of the catalyst by passing hydrogen chloride into acetone, as evidence was obtained that interaction occurred under these conditions; for this reason the solution of the catalyst was prepared by adding aqueous hydrogen chloride of known strength to dry acetone. This necessarily introduced a small quantity of water, but it was never found possible to work with less water than this, for the results were then discordant, owing, doubtless, to changes resulting in condensation of the acetone. Separate tests showed that the concentration of the hydrogen chloride

in the solution used did not change appreciably during the time occupied by an experiment.

The quality of the acetone was found to make an appreciable difference, and it proved necessary to use highly purified material. Concordant results were obtained from acetone, from the bisulphite compound obtained from Kahlbaum (*A*), or prepared and dried by us (*B*); good results attended the use of acetone (*C*) which had been prepared from commercial purified acetone by boiling it with about 3 per cent. of its weight of metallic calcium for some hours, and afterwards fractionating by means of an eight-section Young's dephlegmating column.

The following represent a comparative experiment with the three specimens: water (0.253), alcohol (0.873), and acetic acid (1.143) grams, and the same amount of catalyst being added to three 25 c.c. flasks, which were then filled with acetone at 25°, quantities of 2 c.c. being removed from each and titrated from time to time; x is the titre of the free acetic acid in c.c. of $N/10$ alkali.

<i>A.</i>	$t = 12$	65	107	135	285	$k = 0.0230$
	$x = 14.33$	13.30	12.49	12.00	10.20	
<i>B.</i>	$t = 24$	74	115	142	292	$k = 0.0233$
	$x = 14.33$	13.30	12.59	12.16	10.40	
<i>C.</i>	$t = 30$	80	121	148	302	$k = 0.0243$
	$x = 14.02$	12.86	12.11	11.71	9.80	

It is seen that the results are not completely concordant; for this and similar reasons the authors have not attempted to express all the velocity constants obtained in exactly the same measure. The constants for any one series, however, are strictly intercomparable, the individual experiments being made under identical conditions with only one variant. The different series in acetone are to be regarded as corresponding within a few per cents. only. It would be possible, if it served any really useful purpose, to pass from one series to another by fixing the point at which the conditions of the series in pairs correspond. The interest lies principally in the consideration of the influence of one variant at a time.

The alcohol and acetic acid, which were as nearly as possible anhydrous, were introduced in equimolecular quantities at 25° by carefully standardised pipettes, except when either was a variable in a series, when it was accurately weighed. In all other cases, too, the quantities of the reacting substance were weighed in the flasks, partly filled with acetone, then placed in the thermostat for some time, after which the catalyst in acetone was introduced, and the volume at once made up with pure acetone to 25 c.c., all the liquids having previously attained the temperature of the thermostat. At measured intervals of time, portions of 2 c.c. were withdrawn by means of a carefully

standardised pipette, and titrated with $N/10$ alkali, free from carbonate. In tabulating the titres in each case, that part due to the catalyst was first subtracted." It was ascertained that in the absence of alcohol no measurable diminution in the titre of acetic acid and hydrogen chloride occurred in wet acetone under the experimental condition.

A word must be added with reference to the mode of estimating the initial velocities of change. The titres plotted against time give curves which are not easily expressed by a simple mathematical relationship, so that other methods than the use of formulæ had to be adopted. In the case of the esterification numbers, the curves at first are nearly of the bimolecular form, so that by plotting the reciprocals of the titres against the time, a nearly straight line is obtained, from the slope of which the velocity at the beginning was easily calculated. In the experiments on hydrolysis no such simple relation could be found, and tangents were therefore drawn by hand to the smooth, plotted curves. This method was much less satisfactory than the former, but yields results which are not far from the correct values, and in series VIII the numbers given are identical, because the curves when graphed are indistinguishable in the initial stages.

The function k represents in all cases the diminution of the titre per minute, in c.c., at the commencement (when $t=0$). For comparison, the way in which k changes with the variable is added in each instance.

A. Esterification.

SERIES I.—Constants: Water, 0.253; Acetic Acid, 1.143.

Variant.	Alcohol.	$k \times 10^{-2}$.					
(a)	0.873	$t = 1$	34	88	117	158	2.80
		$x = 13.85$	12.97	11.73	11.12	10.41	
(b)	1.098	$t = 2$	36	82	111	152	3.51
		$x = 13.84$	12.70	11.43	10.77	9.96	
(c)	1.713	$t = 2$	33	78	107	147	5.40
		$x = 13.74$	12.27	10.62	9.76	8.78	
(d)	2.043	$t = 3$	30	75	104	144	6.51
		$x = 13.86$	12.32	10.47	9.45	8.35	
(e)	2.728	$t = 2$	35	70	100	139	8.27
		$x = 13.74$	11.51	9.81	8.70	7.52	
(f)	4.566	$t = 2$	29	64	98	133	12.8
		$x = 13.64$	11.20	8.90	7.45	6.29	
		a.	b.	c.	d.	e.	f.
	$k \times 10^{-2}$ alcohol	3.20	3.19	3.15	3.18	3.03	2.80

SERIES II.—*Effect of Alcohol on the Initial Rate of Esterification of Phenylacetic Acid by Alcohol in Ethereal Solution. Constants: Phenylacetic Acid, 2.00; Ether to 50 c.c.*

Alcohol.		$k \times 10^{-2}$					
(a)	2 c.c.	$t = 4$	14	26	34	44	56
		$x^* = 13.84$	13.16	12.22	11.70	11.10	10.48
(b)	4 c.c.	$t = 3$	14	19	27	37	49
		$x^* = 13.89$	12.33	11.84	11.10	10.38	9.62
		$k \times 10^{-2}$		a.	b.		
		alcohol		4.6	4.6		

* These titres include the hydrogen chloride present as catalyst.

SERIES III.—*Effect of Methyl Alcohol on the Initial Rate of Esterification of Acetic Acid in Ethereal Solution. Constants: Acetic Acid 3.72; Ether to 100 c.c.*

Methyl alcohol.		$k \times 10^{-2}$					
(a)	10 c.c.	$t = 6$	17	27	45	107	
		$x = 30.55$	29.06	27.50	26.30	22.30	13.9
(b)	5 c.c.	$t = 9$	20	34	47	109	
		$x = 30.90$	30.30	29.30	28.60	26.15	5.5
		$k \times 10^{-2}$		a.	b.		
		methyl alcohol		1.3	1.1		

SERIES IV.—*Constants: Water, 0.253; Alcohol, 0.873.*

Acetic acid.		$k \times 10^{-2}$					
(a)	2.738	$t = 32$	86	115	197	271	475
		$x = 33.74$	31.50	30.41	27.88	27.03	
(b)	2.223	$t = 29$	58	111	194	267	450
		$x = 27.31$	26.15	24.35	22.59	21.43	
(c)	1.840	$t = 26$	55	110	192	266	391
		$x = 22.86$	22.01	20.24	18.52	17.42	
(d)	1.473	$t = 21$	52	105	190	264	318
		$x = 18.54$	17.62	16.32	14.80	13.79	
(e)	1.124	$t = 17$	51	104	188	263	245
		$x = 14.19$	13.36	12.41	11.11	10.30	
(f)	0.721	$t = 15$	49	100	184	261	172
		$x = 9.29$	8.80	8.06	7.18	6.54	
		$k \times 10^{-2}$		a.	b.	c.	d.
		acetic acid		1.72	2.03	2.12	2.16
						e.	f.
						2.21	2.48

SERIES V.—*Constants: Alcohol, 0.873; Acetic Acid, 1.143.*

Water.							$k \times 10^{-2}$.
(a)	0.253	$t = 16$	49	94	143	240	2.60
		$x = 14.38$	13.67	12.70	11.82	10.57	
(b)	0.490	$t = 20$	49	94	144	240	2.00
		$x = 14.45$	13.89	13.21	12.51	11.48	
(c)	0.733	$t = 24$	50	95	144	241	1.38
		$x = 14.57$	14.17	13.67	13.12	12.28	
(d)	0.985	$t = 23$	51	97	146	243	0.87
		$x = 14.71$	14.50	14.10	13.73	13.09	
(e)	1.426	$t = 24$	51	98	244		0.57
		$x = 14.85$	14.63	14.47	13.84		
(f)	2.172	$t = 26$	54	100	246		0.34
		$x = 14.81$	14.77	14.59	14.25		
		$a.$	$b.$	$c.$	$d.$	$e.$	$f.$
Water $\times k \times 10^{-2}$		0.66	0.98	1.01	0.85	0.82	0.74

SERIES VI.—*Effect of Water on the Initial Rate of Esterification of Acetic Acid in Absolute Alcohol. Constants: Acetic Acid, 10 c.c.; Alcohol to 50 c.c.; HCl about N/100.*

Water						
(a)	None	$t = 1.5$	7	12	18	28
		$x = 13.62$	12.97	12.52	12.14	11.51
(b)	0.5 c.c.	$t = 1$	6	12	18	25
		$x = 13.74$	13.33	13.01	12.84	12.52
(c)	1.0 c.c.	$t = 1$	7	13	23	
		$x = 13.76$	13.53	13.32	13.05	

For estimations, 5 c.c. of the solution were added to aqueous sodium acetate, the whole being made up to 25 c.c. with water; portions of 1 c.c. were then titrated, and the mean results are those given.

The depressions here noticed as water is added or formed are greater than could be accounted for on the assumption that water acts by hydrating the acetic acid, unless it be supposed that one molecule of water would render several molecules of carboxylic acid inactive.

SERIES VII.—*Constants: Water, 0.253; Alcohol, 0.873; Acetic Acid 1.143.*

Ethyl acetate.						$k \times 10^{-2}$.
(a)	0.182	$t = 17$	79	134	278	2.53
		$x = 14.60$	13.20	12.26	10.56	
(b)	0.310	$t = 28$	80	136	279	2.40
		$x = 14.22$	13.19	12.26	10.58	
(c)	0.715	$t = 27$	79	135	277	2.29
		$x = 14.23$	13.24	12.40	10.81	
(d)	1.028	$t = 28$	80	136	279	2.13
		$x = 14.30$	13.30	12.48	10.97	

SERIES VII.—*Constants: Water, 0.253; Alcohol, 0.873; Acetic Acid, 1.143 (continued).*

Ethyl acetate.						$k \times 10^{-2}$
(e)	1.425	$t = 29$	81	137	260	
		$x = 14.32$	13.38	12.59	11.24	1.98
(f)	2.004	$t = 29$	84	140	283	
		$x = 14.40$	13.49	12.79	11.54	1.82
(g)	2.158	$t = 39$	85	141	284	
		$x = 13.96$	13.27	12.59	11.44	1.75

B. Hydrolysis.

SERIES VIII.—*Constant: Ethyl Acetate, 1.666.*

Water.						$k \times 10^{-2}$
(a)	0.253	$t = 42$	84	134	206	
		$x = 0.27$	0.56	0.83	1.18	0.72
(b)	0.536	$t = 44$	85	136	208	
		$x = 0.35$	0.62	0.95	1.39	0.70
(c)	0.788	$t = 45$	85	136	208	
		$x = 0.33$	0.59	0.92	1.36	0.70
(d)	1.010	$t = 47$	86	137	209	
		$x = 0.35$	0.59	0.88	1.29	0.79
(e)	1.305	$t = 48$	90	140	210	
		$x = 0.34$	0.55	0.77	1.16	0.79
(f)	1.723	$t = 50$	95	140	210	
		$x = 0.29$	0.54	0.74	1.06	0.79
(g)	2.536	$t = 52$	88	143	211	
		$x = 0.26$	0.45	0.65	0.96	0.61

SERIES IX.—*Constants: Ethyl Acetate, 1.666; Water, 0.341.*

Alcohol.						$k \times 10^{-2}$
(a)	None	$t = 54$	132	220		
		$x = 0.42$	0.96	1.46		0.79
(b)	0.431	$t = 55$	142	231		
		$x = 0.38$	0.90	1.33		0.74
(c)	0.806	$t = 57$	146	233		
		$x = 0.34$	0.83	1.21		0.64
(d)	1.154	$t = 58$	147	234		
		$x = 0.37$	0.83	1.15		0.64
(e)	1.608	$t = 62$	150	236		
		$x = 0.31$	0.74	1.04		0.58
(f)	1.942	$t = 63$	152	237		
		$x = 0.35$	0.73	0.98		0.58
(g)	2.718	$t = 64$	153	238		
		$x = 0.33$	0.63	0.86		0.52

SERIES X.—*Constant: Water, 0.339.*

Ethyl acetate.							$k \times 10^{-2}$.
(a)	1.066	$t = 53$ $x = 0.26$	89 0.41	131 0.49 (?)	196 0.82		0.58
(b)	1.381	$t = 36$ $x = 0.21$	69 0.41	133 0.75	177 0.96		0.73
(c)	1.833	$t = 49$ $x = 0.41$	83 0.63	124 0.90	191 1.37		0.99
(d)	2.282	$t = 32$ $x = 0.31$	65 0.63	135 1.22 (?)	169 1.57		1.18
(e)	2.806	$t = 44$ $x = 0.54$	80 0.97	126 1.46	186 2.00		1.46
(f)	3.225	$t = 28$ $x = 0.41$	62 0.88	140 1.73 (?)	165 2.11		1.64
(g)	3.576	$t = 41$ $x = 0.67$	76 1.22	133 1.85 (?)	182 2.62		1.82
		a.	b.	c.	d.	e.	f.
		0.54	0.53	0.54	0.52	0.52	0.51
	$k \times 10^{-2}$ ethyl acetate						0.49

Much of the expense incurred during this work was defrayed from a grant awarded by the Government Grant Committee of the Royal Society, for which the authors desire to express their indebtedness.

GOLDSMITHS' COLLEGE,
NEW CROSS, S.E.

CCXVII.—*Tellurium Dicyanide.*

By HERBERT EDWIN COCKSEGE.

ALTHOUGH cyanides of both sulphur and selenium have been known for some time, the possible existence of a tellurium compound appears not to have been investigated.

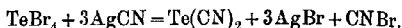
It was found that a cyanide of tellurium can be prepared by double decomposition between silver cyanide and tellurium tetrabromide, the reaction taking place in a suitable organic solvent.

The tellurium tetrabromide was obtained by the cautious addition of bromide to powdered tellurium, the excess of bromine being removed by a current of dried nitrogen while the flask containing the substance was warmed.

The details of a preparation are as follows: The tellurium tetrabromide (35 grams), with twice the required quantity of well-dried

silver cyanide (100 grams), was placed in a round-bottomed flask fused to a reflux condenser, and about 200 c.c. of purified and dried benzene added. The flask was heated to about 90° in a water-bath continuously, with occasional shaking, for three days, when the benzene, in which the tellurium cyanide is not appreciably soluble, was decanted from the silver salts.

It was found to contain much cyanogen bromide, and the reaction may therefore be written :



The residue was then digested with dry ether, which dissolved out the tellurium cyanide, forming a colourless solution.

Inasmuch as the tetrabromide forms a deep yellow solution in ether, the absence of colour indicated that the reaction was complete. After evaporating the ethereal solution at the ordinary temperature, access of moist air being avoided, colourless crystals were obtained, which lost ether on warming; the substance proved to be a compound of ether and tellurium dicyanide, $2\text{Te}(\text{CN})_2 \cdot (\text{C}_2\text{H}_5)_2\text{O}$. Analysis gave:

Found, N = (i) 13.2 and (ii) 13.3.

Te = (i) 58.2, (ii) 58.8, and (iii) 59.8.

$2\text{Te}(\text{CN})_2 \cdot (\text{C}_2\text{H}_5)_2\text{O}$ requires N = 13.0 and Te = 58.9 per cent.

It is known that the tetrachloride and tetrabromide of tellurium form similar compounds with ether.

To prepare the dicyanide, $\text{Te}(\text{CN})_2$, the ethereal solution obtained above was filtered into a distilling flask, and evaporated to dryness from a water-bath, the last traces of ether being removed by a current of dried hydrogen. The cyanide so produced retained the shape of the original crystals, but was grey, owing to a slight coating of tellurium.

The pure cyanide was obtained by distilling this product in a vacuum, about half of the substance being lost in the process as tellurium and cyanogen. The sublimed crystals were analysed :

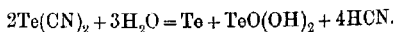
0.1571 gave 0.1114 Te. Te = 70.9.

0.2218 „ 0.0640 cyanogen. (CN) = 28.8.

$\text{Te}(\text{CN})_2$ requires Te = 71.0 ; (CN) = 29.0 per cent.

When exposed to the air, the cyanide in a few minutes assumes the appearance of graphite, owing to deposited tellurium.

With water and alkalis, immediate hydrolysis occurs, and tellurium is precipitated in the form of black flocks. The course of the hydrolysis is similar to that of the dichloride and dibromide of tellurium :



When heated in air, the cyanide burns with a pale blue flash.

The decomposition of the compound into tellurium and cyanogen

takes place to a slight extent at 100°, but more rapidly as the temperature rises; there is a sudden increase in the rate of evolution of the gas at about 190°, at which temperature partial sublimation of the cyanide occurs.

The substance is slightly soluble in chloroform or carbon tetrachloride; methyl alcohol dissolves it in the cold without change, but hydrolysis occurs when the solution is warmed.

At the ordinary temperature, one gram of the substance requires about 60 c.c. of ether for solution.

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OXFORD.

CCXVIII.—*Boron Thiocyanate.*

By HERBERT EDWIN COCKSEGE.

THE thiocyanates of phosphorus and silicon were first described by Miquel (*Ann. Chim. Phys.*, 1877, [v], 11, 343); they have since been investigated by A. E. Dixon (*Trans.*, 1901, 79, 541) and J. E. Reynolds (*ibid.*, 1906, 89, 397).

The corresponding boron compound was alluded to by Miquel, in the paper mentioned above, as follows: "Finally, after experiments as yet incomplete, I can assert that boron bromide reacts similarly with lead sulphocyanide to give a sulphocyanide with properties analogous to those of the silicon compound."

Boron bromide reacts with silver thiocyanate with the formation of boron thiocyanate, which is extracted from the product by means of cold benzene.

The boron bromide was prepared according to Gattermann's method (*Ber.*, 1889, 22, 195) by the action of bromine on crude boron, obtained by the action of magnesium powder on anhydrous borax. The final distillation was carried out with care, since the mercuric bromide formed after decolorisation of the crude product with mercury is carried over in appreciable quantities in the last portion of boron bromide vapour.

To prepare the boron thiocyanate, a small bulb of boron bromide was placed together with twice the theoretical quantity of thoroughly dried silver thiocyanate in a well-stoppered bottle, pure benzene added, and the bulb broken by vigorous shaking. The shaking was continued for a few minutes, after which the colourless solution of the thiocyanate was filtered, and the benzene removed at the ordinary

temperature by means of a current of dry air. The substance was deposited in glistening, colourless crystals, and a further quantity could be obtained by extracting the residual silver salts with benzene.

Traces of hydrolysis occur unless extreme care is taken, the crystals becoming clouded and discoloured as the solution becomes concentrated. Analysis gave :

B = (i) 5.8, (ii) 6.3, and (iii) 5.8.

N = (i) 21.6 and (ii) 22.3.

S = (i) 51.6 and (ii) 51.8.

$B(SCN)_3$ requires B = 5.9 ; N = 22.7 ; S = 51.9 per cent.

The substance separates from benzene in short, rhombic crystals, or sometimes in the form of radiating needles. It crystallises from ether in thin, colourless plates.

When heated on a platinum wire in the Bunsen flame, the thiocyanate burns, leaving a charred residue, which, on ignition, gives boron nitride. In the absence of air, it is largely destroyed on heating, only a small portion distilling unchanged.

At 100° the substance darkens considerably, and a benzene solution deposits brown flocks when it is boiled.

When exposed to moisture or treated with alkalis, the thiocyanate is at once hydrolysed to boric and thiocyanic acids, a solution of ferric chloride giving an intense coloration.

With the view of determining whether the substance possesses the constitution of a thiocyanate, $B(SCN)_3$, or of a thiocarbimide, $B(NCS)_3$, its behaviour towards aniline was investigated.

A. E. Dixon has shown (*loc. cit.*) that the corresponding phosphorus compound acts as a thiocarbimide towards the base, yielding the corresponding thiocarbamide, whereas from the work of J. Emerson Reynolds the silicon compound exhibits the behaviour of a thiocyanate.

A solution of the boron compound in benzene was added to a concentrated solution of aniline in the same solvent. Some heat was evolved, and a mass of white crystals was precipitated. These were washed by decantation with benzene, drained, and dried, access of moisture being avoided as much as possible.

The crystals contained no boron, but gave reactions for aniline and a thiocyanate; they melted at about 78°, softening before that temperature was reached. The substance gave no thiocarbamide reactions when freshly prepared, but after boiling with water, or allowing it to stand for some time, a mirror of lead sulphide was obtained on warming a portion with an alkaline solution of lead.

The substance had in fact been converted into phenylthiocarbamide,

which melts at 154° ; a specimen prepared from the original crystals melted at 153° . On analysis:

0.0914 gave 14.2 c.c. N_2 (moist) at 11° and 756 mm. $N = 18.6$.

0.4136 " 0.6679 $BaSO_4$. $S = 22.2$.

$C_4H_8N_2S$ requires $N = 18.4$; $S = 21.1$ per cent.

The above experiments thus indicate that the boron compound in solution behaves as a thiocyanate rather than as a thiocarbimide.

The boron remains in solution in the benzene probably as the anilide of boron, but it was not isolated as such; the anilide, which does not seem to have been described, is extremely soluble in benzene, and can only be obtained from a solution in that solvent by concentrating and keeping the resulting gum for some time.

In conclusion, I wish to express my best thanks to Dr. Baker for the helpful interest he has taken in the above work.

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OXFORD.

CCXIX.—*The Viscosity of Fuming Sulphuric Acid.*

By ALBERT ERNEST DUNSTAN and ROBERT WILLIAM WILSON.

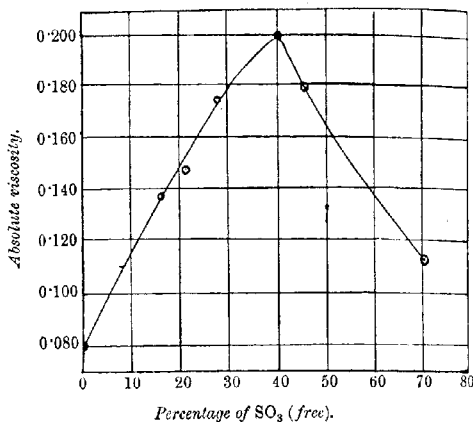
In a recent paper (Trans., 1907, 91, 83), the present authors published a viscosity-concentration curve for aqueous solutions of sulphuric acid. This curve was shown to follow very closely that originally determined by Knietzsch (*Ber.*, 1901, 34, 4069), and it was pointed out that the investigation clearly indicated very considerable association between the acid and the water.

A well-defined maximum point was found to exist at a concentration of 85 per cent. sulphuric acid, corresponding with the well-known compound $SO(OH)_4$, and a minimum point at the concentration 95 per cent. sulphuric acid, which corresponds with the hydrate $3H_2SO_4 \cdot H_2O$. Now it appears probable that the compound $SO(OH)_4$ is present in solution in the aggregate $[O:S(OH)_4]_n$, where n is a whole number of considerable magnitude, and similarly, at the concentration indicated by the minimum point there is present the aggregate $[3H_2SO_4 \cdot H_2O]_m$, where m is less than n , since the exceedingly high value at these two points of the viscosity coefficient, 0.94794 and 0.83255 respectively, points to the existence of complexes of great molecular weight.

In the above-mentioned paper, the curve ended at 100 per cent. sulphuric acid, with evidence that the viscosity would be found still increasing with a greater percentage of the anhydride.

Owing to the kindness of Dr. Messel, to whom the authors take this opportunity of expressing their indebtedness, an ample supply of very pure acids of varying content of free sulphur trioxide was placed at their disposal.

The solidity of some of the specimens necessitated the work being carried out at a higher temperature than 25° , at which the previous experiments had been conducted; hence 60° was taken as the constant temperature. The extreme manipulative difficulty of working with these strongly fuming liquids, and the remarkable way in which they absorbed water, even during the progress of the determinations, precluded the attainment of any high degree of accuracy, but, for pur-



poses of comparison, it must be borne in mind that each point was determined under precisely similar conditions.

Knietsch (*loc. cit.*), using a variety of methods, found discontinuities in the curve connecting concentration with physical properties at the following points:

Melting-point determinations: $2\text{H}_2\text{SO}_4, \text{H}_2\text{O}$, $4\text{H}_2\text{SO}_4, \text{SO}_3$, $\text{H}_2\text{SO}_4, 2\text{SO}_3$, $\text{H}_2\text{SO}_4, \text{H}_2\text{O}$.

Viscosity: $\text{H}_2\text{SO}_4, \text{H}_2\text{O}$, $\text{H}_2\text{SO}_4, \text{SO}_3$.

Conductivity: $2\text{H}_2\text{SO}_4, \text{H}_2\text{O}$, $\text{H}_2\text{SO}_4, \text{H}_2\text{O}$, and 15 per cent. SO_3 .

In the present work (see figure), it is to be noticed that the viscosity concentration curve rises sharply from pure sulphuric acid to the acid containing 40 per cent. of sulphur trioxide, at which concentration the compound $\text{H}_2\text{SO}_4, \text{SO}_3$ probably exists. This maximum also is in substantial agreement with Knietsch's work. At this

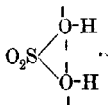
point the viscosity coefficient attains the dimensions 0.2045, whereas for water at 60° is 0.00464, and for sulphuric acid, 0.0832.

Per cent. free SO ₃	Viscosity.	Per cent. free SO ₃ .	Viscosity.
70.0	0.1147	21.5	0.1488
46.0	0.1791	16.3	0.1383
40.6	0.2045	0.0	0.0832
27.72	0.1753	Water at 60°	0.00464

From the maximum, the curve rapidly falls, and as free sulphur dioxide is approached, the viscosity falls to the same dimensions as that of pure sulphuric acid.

The general conclusions to be drawn from this work are :

- (1) The high degree of association of sulphuric acid, due probably to each hydroxyl group functioning as the seat of very considerable residual affinity, thus :



- (2) Further evidence as to the existence, in solution, of molecular aggregates, such as H₂SO₄.SO₃.

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CCXX.—*The Densities of Krypton and Xenon.*

By RICHARD B. MOORE, B.Sc.

THE volume of krypton and xenon isolated by Ramsay and Travers during their classical research on the rare gases of the atmosphere amounted to 12 c.c. of krypton and 3 c.c. of xenon. The two best density determinations obtained by them for krypton were made on two samples of gas, one of which had been fractionated from argon and the other from xenon. The former gave a density of 40.82 and the latter 40.73. Later, Ramsay, starting with 191.1 kilos. of air, obtained 7.5 c.c. of krypton and 0.87 c.c. of xenon. This krypton gave a density of 40.81. To make an accurate density determination with the original 3 c.c. of xenon was by no means easy, and the two highest figures obtained, 63.64 and 64.0, agreed exceedingly well in the circumstances. The density of xenon has generally been taken as 64.0, and its atomic weight, on the assumption that it is a monatomic gas, as 128.0 (*Phil. Trans.*, 1901, 197, A, 66).

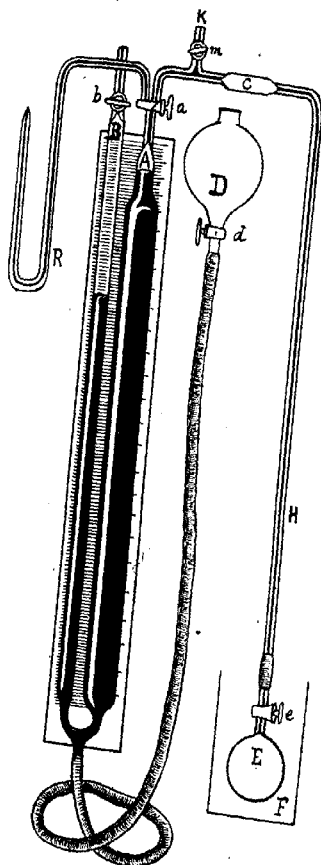
Owing to the small volume of gas available for fractionation, it is probable that the krypton in the above experiments contained small traces of argon, and that the xenon also was not entirely free from krypton. This source of error would give low results for the densities of both gases. In addition, the volume of the bulb used was only 7 c.c. Recently, during a search for possible new elements in the atmosphere (*Proc. Roy. Soc.*, 1908, **81**, 195), the author fractionated in Sir William Ramsay's laboratory the residues from 120 tons of liquid air, which gave krypton and xenon in sufficient quantities to make it possible to obtain pure samples of both gases.

The density apparatus used was a slight modification of that designed by Ramsay and Travers (*Phil. Trans.*, 1901, **A**, 197, 54), and is shown in Fig. 1. It consists of a U-shaped gas burette fastened to a strip of mirror glass, on the surface of which is etched an accurate millimetre scale. The tube *B* is of the same diameter as the upper portion of *A*, hence a correction for capillarity is unnecessary. *C* contains a roll of silver foil in order to prevent minute globules of mercury, carried over by the gas, from entering the bulb *E*. *K* leads to a Töpler pump. The bulb *E* is connected with the capillary tube *H* by means of a selected piece of pressure tubing wired on. It was found that such a connexion remained perfectly gas-tight during a period very considerably longer than was required for an experiment.

The density bulb used had a volume of 32.7077 c.c. The volume of the sealed counterpoise was the same as that of the bulb. A long-armed Oertling balance sensitive to 0.01 milligram and carefully standardised weights were used. These weights, although standardised relatively to each other, were made absolute, inasmuch as they were used for determining the weight of the bulb full of water. No correction for latitude has been applied.

The method of manipulation was as follows: By raising the globe *D* the mercury was run into the burette up to the stopcocks *a* and *b*. The latter were then closed. The reservoir *D* was then lowered so as to leave a barometric vacuum in the tubes *A* and *B*, after which the stopcock *d* was closed. The density bulb had meanwhile been completely exhausted and carefully weighed. It was then attached to the tube *H*. The vessel *F*, surrounding the bulb, was packed with ground ice, and distilled water, previously cooled almost to zero, was poured in until the level of the water reached the upper surface of the ice. The stopcocks *a* and *m* were then turned, the apparatus thus being put in communication with the pump. In this manner, the air in the bore of the stopcock *a* was completely removed. After exhaustion was complete, *a* and *m* were closed and *d* opened, the mercury rising in the burette. The gas was then run into the burette through the capillary tube *R*, which was completely filled with mercury. On

turning the stopcocks *a* and *e* so as to put *A* into communication with *E*, the gas entered the bulb. The stopcock *b* was then opened and the reservoir *D* adjusted so that the level of the mercury in *A*



lay close to the top of the tube. After half an hour, the pressure was observed, the stopcock *e* closed, and the barometer read. The gas contained in *C* and *A* was removed through the pump, and the globe

was suspended on the balance. Owing to the small size of the globe, no correction was made for shrinkage under atmospheric pressure.

Xenon.—The xenon used in the density determinations had been separated from the krypton by a long series of fractionations. The gas thus obtained, after it had been sparked with oxygen and the oxygen removed with phosphorus, was condensed in the fractionating bulb at the temperature of liquid air. As krypton has a vapour pressure at this temperature of 17 mm., and the vapour pressure of xenon at the same temperature is only 0.17 mm., the two can be separated by removing the krypton from the xenon by means of a Töpler pump. It was found during the preliminary fractionation that a mixture of solid krypton and xenon could be apparently pumped "dry" and yet some krypton would be retained below the surface of the solid xenon. On vaporising all the gas, however, and re-depositing, this krypton could be pumped off. This process was therefore repeated several times during the final attempt to get rid of all traces of krypton. The gas then pumped off was apparently pure xenon, as its spectrum did not show the slightest traces of the principal krypton lines. Nevertheless, it was rejected as probably containing traces of krypton.

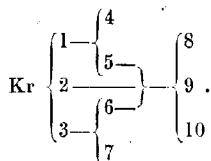
A portion of the pure xenon was then fractionated at -136° by means of light petroleum cooled with liquid air. Four fractions were obtained, and density determinations were made with fractions 2 and 3. The results were as follows:

	Fraction 2.	Fraction 3.	
Volume of density bulb (in c.c.)...	32.7077	32.7077	32.7077
Temperature	0°	0°	0°
Pressure on gas, corrected (in mm.)	480.0	446.4	521.7
Weight (in grams).....	0.12044	0.11223	0.13106
Hence, density (0=16)	65.253	65.380	65.328

As the density of fraction 2 was lower than that of fraction 3, the former probably still contained a very small trace of krypton, and the experiment may therefore be rejected. It is probable that this trace was contained in the first portion of fraction 2, and as the whole volume of the fraction was 40 c.c., we may assume that any krypton in fraction 3 would have no effect on its density within the limits of experimental error. The mean of the two determinations on fraction 3 (65.35) may therefore be taken as the density of xenon.

Krypton.—It is easier to obtain pure xenon than pure krypton. In the former case it is only necessary to free the gas from krypton. In the latter, both argon and xenon must be removed. In the fractionation of a mixture of three gases, it is always easier to obtain pure samples of the gases which possess the lowest and highest boiling points than it is to obtain a similar sample of the gas with an inter-

radiate boiling point. Consequently, special pains were taken to purify the krypton. The gas obtained during the progress of the work already referred to had been repeatedly fractionated, and its spectrum showed none of the argon or xenon lines. In order to be sure that the krypton was free from these gases, it was refractionated at the temperature of liquid air, according to the following plan:



Fractions 1 and 3 were refractionated separately, fractions 4 and 5 being rejected as containing either argon or xenon; 2, 5, and 6 were mixed and refractionated, 8 and 10 being rejected, whilst fraction 9, which contained most of the gas, was considered as being practically pure krypton.

This gas was then fractionated at -130° , a bath of light petroleum mixed with liquid air being used. Ten fractions were thus obtained. As the gas was more likely to be contaminated with traces of argon than with traces of xenon, fractions 8 and 9 were selected for density determinations. As the volume of No. 8 was not quite large enough, a small portion of No. 7 was added. The gas samples were sparked with oxygen over sodium hydroxide solution, and the excess of oxygen was removed by phosphorus.

During the early stages of the separation of the mixed rare gases, it was found that they were not only contaminated with oxygen and nitrogen, but also with traces of hydrocarbon vapour, derived from the pentane used in lubricating the compressors of the liquid-air plant. The mixed gases had therefore been passed twice heated over copper oxide, but on sparking the samples of krypton obtained as described above, it was found that some of the hydrocarbon vapour had escaped oxidation by the copper oxide. This, however, did not vitiate the value of the fractionation from argon and xenon. The last trace of the hydrocarbon was, of course, removed by the sparking. Two determinations were therefore made on fraction 8, with the following results:

Fraction 8.

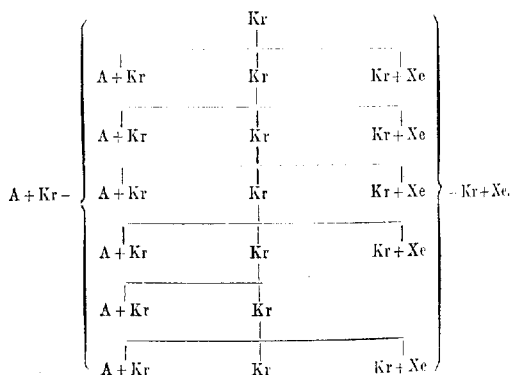
Volume of density bulb (in c.c.)	32.7077	32.7077
Temperature	0°	0°
Pressure on gas, corrected (in mm.).....	474.0	478.3
Weight (in grams)	0.07566	0.07633
Density.....	41.509	41.500

As the gas was taken through the pump after the first determination there was a possibility of its being contaminated with a very small trace of air. This would make the second result low, and 41.509 may be accepted, therefore, as being the more correct figure.

A determination on fraction 9 gave a low result, probably due to insufficient sparking. At this stage of the work, the author was unfortunately forced to leave England, and Sir William Ramsay and Mr. A. T. Cameron very kindly offered to make another density determination with this fraction of the gas. After prolonged sparking and removal of oxygen, the density obtained was 39.53. It was difficult to understand why fraction 9 should have a lower density than fraction 8. They therefore thoroughly sparked fractions 5 and 6, and mixed the resulting gas with No. 9. The whole was then fractionated once at liquid air temperature, all that could be readily pumped off constituting fraction 1. The remainder of the gas was taken off in two fractions (2 and 3), the middle fraction being much the larger. A determination on this gave the following result:

Volume of density bulb (in c.c.)	32.7077
Temperature	0°
Pressure on gas, corrected (in mm.)	765.9
Weight (in grams)	0.11833
Density	41.18

They then decided to make one last and extremely thorough attempt to obtain a fraction with a density as high as 41.5. The spark spectrum led them to suspect argon in the first and middle fractions, whilst No. 3 seemed to show traces of xenon. The gas was therefore refractionated according to the following scheme. In each case the middle fraction was 19/30ths or more of the whole :



Each time the first fraction was withdrawn, through the pump, the second was allowed to run into a mercury reservoir under slightly reduced pressure, whilst the gas which was left in the fractionating bulb after equilibrium was established constituted fraction 3. The final krypton fraction had not therefore been passed through the pump at any stage of the fractionation, which practically eliminated contamination with air.

The results obtained were as follows :

Volume of density bulb (in c.c.)	32.7077
Temperature	0°
Pressure on gas, corrected (in mm.)	772.5
Weight (in grams)	0.12329
Calculated density	41.504

They considered this figure correct to one part in 2,000. The density of krypton may be therefore taken as the mean of 41.504 and .509, namely, 41.506. On the assumption that krypton and xenon are monatomic gases, their atomic weights would therefore be 83.012 and 130.70 respectively. These new figures do not throw them out of place in the periodic table.

I desire to thank Sir William Ramsay and Mr. A. T. Cameron for their independent work which they so kindly did, and which constituted a rigorous confirmation of my own result; also the former for his many helpful suggestions.

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CXXI.—*An Examination of the Conception of Hydrogen Ions in Catalysis, Salt Formation, and Electrolytic Conduction.*

By ARTHUR LAPWORTH.

Part I.

With E. Fitzgerald the present author has shown that the addition of water to a liquid, such as alcohol, containing an acid leads to a reduction in the number of hydrogen ions * present, and has found that the retardation of esterification and ester hydrolysis by water cannot be explained by hydration of the intermediate products in the esterifi-

* The term "hydrogen ion" refers throughout to the non-hydrated ion, or, in other words, to that supposed to be responsible for hydron catalysis.

cation process. The Arrhenius view of the way in which water hydrolyses the salts of weak bases was also found to be inadequate to the case.

To obtain quite convincing evidence from esterification experiments as to the non-validity of the latter assumption would be extremely difficult, owing to complication arising from the large number of substances present. Experimental attack on a different line was necessary, and it was therefore sought to determine, first, whether hydron catalyses, other than those already known, were affected by water in a similar manner; secondly, if so, whether it would be possible to obtain for examination a case in which the amount of water compared with that of the substance catalytically changed might be made very small.

The Transformation of Hydrazobenzene into Benzidine in Alcoholic Solution.

This well-known change, which is brought about by acids, was especially interesting, because here the substance changed is not a carbonyl compound.

A solution of this substance in absolute alcohol was divided into quantities of 10 c.c. in test-tubes containing respectively 0, 0.2, and 0.4 c.c. of water. To each was then added 5 c.c. of absolute alcohol to which a little sulphuric acid had been added. Benzidine sulphate began to appear in the first tube in about five seconds, in the next in forty seconds, and in the last in eighty seconds. This observation was frequently repeated under similar conditions, with results of the same nature, showing that the effect of water on the catalysis of hydrazobenzene in absolute alcohol is of the same order as its effect on catalysed esterification; the speed of change over a wide range is about inversely as the amount of water present. Fairly concordant results are obtained if a standard tube is used to ensure a definite degree of opalescence at the point timed.

The Bromination of Ketones.

The bromination of carbonyl compounds has already been shown to be catalytically accelerated by mineral acids, and in the case of acetone the speed of the disappearance of the bromine is a measure of a change in the ketone itself (Trans., 1904, 85, 31, *et seq.*).

Solutions of a number of ketones, including acetone, menthone, and acetophenone in alcohol, were acted on by bromine in alcoholic solution with hydrogen chloride as catalyst. In all cases the speed with which a given quantity of bromine disappeared was nearly inversely

proportional to the concentration of water present when the quantity of water was more than one per cent. of the total weight.

The following widely different cases of hydrogen ion catalyses in alcohol had thus been shown to be affected by water in a similar manner, namely, the decomposition of diazoacetic ester (Bredig and Fraenkel, *Ber.*, 1906, **39**, 1756, *et seq.*), esterification of carboxylic acids (Goldschmidt), the change of hydrazobenzene into benzidine, and the bromination of ketones. This suggested that a common explanation of them all is to be looked for, but hydration of the substance undergoing catalysis still remained a possible, although a somewhat remote, one.

The bromination of acetone appeared to offer a means of deciding this point, for here an enormous excess of the catalysed compound as compared with the water could be employed. It has been shown that the speed of disappearance of the bromine is a measure of the speed of a change in the acetone (compare *Trans.*, 1904, **85**, 31).

Influence of Small Quantities of Water on the Velocity of Bromination of Acetone without Solvent.

A quantity of anhydrous acetone (*A*) was mixed with a little dry bromine, and another quantity (*B*) with a few drops of sulphuric acid. After placing these in a thermostat at 25° for some time, portions of 25 c.c. of *A* were then measured into 50 c.c. flasks containing varying quantities of water, also kept immersed in the thermostat, and subsequently 25 c.c. of *B* were added, and the time elapsing between its addition and the complete disappearance of the bromine was taken. The following results were obtained; the numbers given under in the third line are obtained by assuming the water value 0.10 for 50 c.c. of acetone, a number calculable from the first and last observation.

The experimental error in the time observation amounts perhaps to about 10 seconds:

Water	=	0	0.1	0.25	0.5	0.75	1.00 grams
Time	=	45	85	155	265	400	510 secs.
Calc.	=	—	93	164	278	394	—

A number of other experiments with this and other specimens of acetone gave similar results, but different specimens differed slightly in their water values, as was to be expected, since traces of moisture so greatly influence the speed. Experiments on a smaller scale with acetone, which had been prepared from the bisulphite compound and purified with every care, gave results of the same order.

Catalysis in Other Solvents and Possible Impurities in the Water.

Phenomena very similar to those observed in alcohol or acetone are noticed in other oxygenated organic media, and marked retardation by water of acid catalysis occurs to a greater or less extent in ether, ethyl acetate, and other common liquids which dissolve water. The different extent to which these respond is dealt with elsewhere.

The suspicion that the effects noticed are due to impurities in the water may be dismissed at once; these effects are so considerable that in order that they might be caused by, say, an alkali, it would be necessary to suppose the water to contain impossible quantities of this.

Mathematical Treatment of the Question.

Probably the main difficulty in obtaining acceptance of the view, that the influence of water on catalysis by acids is accounted for by a reduction in the number of hydrogen ions, or, in other words, by a diminution of the available acid, lies in the initial difficulty in demonstrating that the conception is in accordance with the generalisations of Ostwald and his school, and in showing that it can be applied to the facts as readily as the original conception of Arrhenius, which, in its first form, is not consistent with the phenomena of acid catalysis. For this reason, and for the further consideration of various aspects of the question, it will be necessary to develop, on strict lines, and first assuming the presence of hydrogen ions as the active catalysts, an expression for the state of a monobasic acid, HR , in a mixture of basic liquids. Representing the amounts of each liquid by the symbols W, W' , etc. respectively, those of their complexes with hydriions as I, I' , etc., and the corresponding undissociated compounds as S, S', S'' , etc., the following expressions represent the state of equilibrium. No allowance in the general expressions need be made for a possible union between the bases, as this would simply result in the introduction of more constituents of the series W , with corresponding additions to the series S and I . The symbols S, S' , etc. include the acid united with the solvents whether ionisable or not, and R represents the total anions of the acid whether united to the liquids W, W' , etc., or not. V is the volume occupied by the system, and A is the original amount of acid. All quantities are expressed in gram-equivalents. Naturally, K, K_2 , etc. may be expected to vary with an alteration in the proportions of the solvent constituents, but will otherwise be constant.

$$(1) \quad HR = \frac{H.R}{K_1 V'}$$

$$(2) \quad I = \frac{H.W}{K_2 V'}; \quad (2') \quad I' = \frac{H.W'}{K_2 V'}; \text{ etc.,}$$

$$(3) \quad S = \frac{I.R}{K_1 V'}; \quad (3') \quad S' = \frac{I'.R}{K_1 V'}; \text{ etc.,}$$

so

$$(4) \quad H + HR + \Sigma I + \Sigma S = A \quad (\text{where } \Sigma I = I + I' + \text{etc.}).$$

$$(5) \quad HR + R + \Sigma S = A \quad (\text{where } \Sigma S = S + S' + \text{etc.}).$$

Whence may be derived:

$$(6) \quad \text{from (4) and (5), } \Sigma I + H = R.$$

$$(7) \quad \text{from (2) and (2'),}$$

$$I' = \frac{W'.K_2}{K_2 V'} \quad \text{Hence } \Sigma I = I \frac{K_2 \Sigma W}{W K_2},$$

where

$$\Sigma \frac{W}{K_2} = \frac{W}{K_2} + \frac{W'}{K_2} + \text{etc.};$$

$$(8) \quad \text{from (3),}$$

$$S' = I' \frac{W'.K_2.R}{K_2 V'.K_2 V'} \quad \text{Hence } \Sigma S = I.R. \frac{K_2 \Sigma W'}{W.K_2 V'}$$

or, putting $\frac{W}{V}$ = active mass, M , etc.

from (2),

$$\Sigma S = R. \frac{H}{V} \Sigma \frac{M}{K.K_2},$$

where

$$\Sigma \frac{M}{K.K_2} = \frac{M}{K.K_2} + \frac{M'}{K'.K_2} + \text{etc.};$$

$$(9) \quad \text{but from (6), (7), and (2),}$$

$$R = \frac{H.W}{K_2 V} \left[\frac{K_2 \Sigma W'}{W K_2} \right] + H \\ = H \left[\Sigma \frac{M}{K_2} + 1 \right].$$

$$(10) \quad \text{By combining (5), (1), and (8),}$$

$$R \left[\frac{H}{K_1 V} + 1 + \frac{H}{V} \Sigma \frac{M}{K.K_2} \right] = A.$$

$$(11) \quad \text{and introducing (9),}$$

$$H^2 \left[\frac{1}{K_1} + \Sigma \frac{M}{K.K_2} \right] + HV = \frac{AV}{\Sigma \frac{M}{K_2} + 1},$$

and for 1 gram equivalent of acid,

$$H^2 \left[\frac{1}{K_1} + \Sigma \frac{M}{K.K_2} \right] + HV = \frac{V}{\Sigma \frac{M}{K_2} + 1}.$$

Concentration of Hydrogen Ions at Infinite Dilution.

At infinite dilution the term not containing V must be neglected, (12) and

$$H \frac{A}{\sum \frac{M}{K_2} + 1},$$

or at infinite dilution the hydrogen ions in solutions containing A gram equivalents of all acids have the same concentration, $\sum \frac{M}{K_2}$ being independent of the acid and depending only on the nature of the solvent.

This is in accordance with the well-known generalisation that the molecular activity of all acids as measured by their effect in inverting sucrose and hydrolysing esters tends towards a maximum value which is the same for all acids.*

Relation between Molecular Conductivity and the Number of Hydrogen Ions.

The conductivity is given by the sum of the number of the ions of each kind \times its mobility:

$$\begin{aligned} &= \sum \mu I + \mu_1 H + \nu R \\ &= \mu \frac{H.W}{K_2.V} + \mu' \frac{H.W'}{K_2'.V} + \text{etc.} + \mu_1 H + \nu H \left[\sum \frac{M}{K_2} + 1 \right] \\ (13) \quad &= H \left[\sum \mu \frac{M}{K_2} + \mu_1 + \nu + \nu \sum \frac{M}{K_2} \right], \end{aligned}$$

that is to say, the molecular conductivity is proportional to the number of hydrogen ions multiplied by a constant which depends only on the nature of the solvent. In other words, the relationship between the so-called degree of dissociation of an acid (as measured by its conductivity) and the hydrogen ions is independent of the nature of the acid.

This is in agreement with the generalisations of Ostwald and Arrhenius, who have shown that the power of an acid to hydrolyse methyl acetate, or to invert sucrose, is the same for all acids at the same degree of dissociation as measured by electrical methods.*

* The generalisations, as thus stated, are not strict, but the point insisted on is that the expressions evolved as above, are in agreement with the experimental facts to precisely the same extent as are those derived by the application of the original ionisation theory of acids.

Variation of "Degree of Dissociation" with Volume of Solution.

The degree of dissociation, x , of an acid as at present measured represents the ratio = $\frac{\text{conductivity at volume } V}{\text{conductivity at infinite volume}}$, which, from the last paragraph = $\frac{H \text{ at vol. } V}{H \text{ at infinite dilution}}$.

$$\text{But from (9) above, } H = R \frac{1}{\sum \frac{M}{K_2} + 1},$$

$$\text{hence } x = \frac{R}{\sum \frac{M}{K_2} + 1} \cdot \frac{\sum \frac{M}{K_2} + 1}{R_\infty} = \frac{R}{R_\infty}.$$

$$\text{But } R_\infty = H_\infty \left[\sum \frac{M}{K_2} + 1 \right] = A \text{ (from 11 above),}$$

$$\text{so that } x = \frac{R}{A} \text{ or } R = Ax \text{ (= } x, \text{ when } A = 1).$$

Replacing H in the equation (10) by $\frac{x}{\sum \frac{M}{K_2} + 1}$, and A by 1,

$$\begin{aligned} & \frac{x^2}{\left[\sum \frac{M}{K_2} + 1 \right]^2} \left[\frac{1}{K_1} + \sum \frac{M}{K K_2} \right] + \frac{xV}{\sum \frac{M}{K_2} + 1} = \frac{V}{\sum \frac{M}{K_2} + 1}, \\ (14) \text{ or } & x^2 + x \frac{\sum \frac{M}{K_2} + 1}{\frac{1}{K_1} + \sum \frac{M}{K K_2}} V = \frac{\sum \frac{M}{K_2} + 1}{\frac{1}{K_1} + \sum \frac{M}{K K_2}} V, \end{aligned}$$

that is to say, the foregoing view leads to precisely the same expression as is applied at the present time to the "dissociation" of weak acids as measured by their electrical conductivity, namely,

$$x^2 + xkv = kv,$$

where k is the "dissociation constant."

(15) This gives the dissociation constant of the acid :

$$k = \frac{\sum \frac{M}{K_2} + 1}{\frac{1}{K_1} + \sum \frac{M}{K K_2}},$$

or, in aqueous solution,

$$k = \frac{\frac{M}{K_2} + 1}{\frac{1}{K_1} + \frac{M}{K K_2}},$$

where M is the active mass of liquid water, and K is the ionic dissociation constant of the hydroxonium salt.

It is clear from the preceding formula for k that the more strong basic solvents (where $\frac{1}{K_2}$ is large) will tend to minimise the inherent differences in potency between strong acids, so that it may be anticipated that mineral acids which have nearly equal constants in water will show very considerable divergencies in less basic solvents, such as ether or alcohol.

In order to demonstrate in a simple manner that this must be the case, for the moment let the simplifying assumption be made that the dissociation constant of the hydroxonium salts of strong acids is the same, $= K$.

The above may be written

$$k = \frac{KK_1(M + K_2)}{K_2K + MK_1}.$$

In the case of bases of the strength of water, the hydrogen ions have been shown to be very few, that is, K_2 is very small, and may be neglected in comparison with M .

$$(16) \quad k = \frac{KK_1M}{K_2K + MK_1}.$$

$$(17) \quad K_1 = \frac{KK_2}{M} \left[\frac{k}{K - k} \right].$$

$\frac{KK_2}{M}$ is constant, and the intrinsic acidic affinity or potency of the acid, K_1 , is therefore proportional to $\frac{k}{K - k}$.

Taking two fairly strong acids, A and B , with dissociation constants in water nearly equal to that of salts, say, $0.95K$ and $0.9K$, their relative intrinsic acidity is given by $\frac{0.95}{0.05}$ and $\frac{0.90}{0.1} = 19$ and 9 respectively. That is, whilst their true affinities are in the ratio 2.11, their affinities in water appear to be only in the ratio 1.05.

In respect to this point, Goldschmidt and Sunde have shown (*Ber.*, 1906, 39, 719) that, although picric acid appears nearly as powerful as hydrochloric acid in aqueous solution, yet in alcoholic solution it has only about one-tenth of the catalytic activity of the latter. Similarly, picric acid and trichloroacetic acid differ far more in alcohol than in water, and similarly with di- and tri-chloroacetic acids.

Hydrolysis of Salts in Alcoholic and Similar Media by Water.
Water Value of Solvents.

The effects of adding a small quantity of a relatively strong base to a solution of an acid in a weak base may readily be deduced from the general equation, providing certain conditions are known, as is perhaps the case where water is added to a dilute solution of hydrogen chloride in absolute alcohol.

It is known that in absolute alcohol the acid at $N/10$ dilution exhibits already about 0.4 of its maximum conductivity, and this fraction increases with addition of water to a comparatively small extent, so that for very small additions of water it may at a first approximation be assumed nearly constant. This fraction represents, according to the present conception, the proportion present as anions, so that

$$H = \frac{R}{1 + \frac{M}{K_2} + \frac{M'}{K'_2}} = \frac{K_2 x}{K_2 \left[1 + \frac{M'}{K'_2} \right] + M},$$

where M and K_2 refer to water, and M' and K'_2 to alcohol. Also $R = x$ = measured degree of dissociation, and assuming that the velocity of esterification at any time is proportional to the active masses of the hydrogen ions, of the carboxylic acid, and the alcohol respectively, it is given by the expression (neglecting 1 in comparison with $\frac{M'}{K'_2}$ for the first time):

$$-\frac{du}{dt} = AK_2 p x M' \frac{u}{\frac{K_2}{K'_2} M' + M},$$

where p is a new constant dependent on the nature of the carboxylic acid, and u is the active mass of this acid.

This is in accordance with the observations of Goldschmidt and Udby (*Zeitsch. physikal. Chem.*, 1907, **60**, 735, *et seq.*) for the velocity of esterification of numerous acids by hydrogen chloride in alcohol and varying amounts of water, and their functions have the same significance.*

* For comparison, the expression used by these authors may be given here:

$$\frac{du}{dt} = k \pi b c \frac{a - x}{r + u + x},$$

the corresponding symbols being:

(G. and S.) $k\pi$, b , r , c , $a - x$, $u + x$.

(I.) $p \frac{K'_2}{M'}$, M' , $\frac{K_2}{K'_2} M$, Ax , u , M .

The function c (G. and S.) was shown to be probably proportional to the measured dissociation of the catalyst, $k\pi$ to depend on the nature of the carboxylic acid and of the alcohol, r to be independent of the nature of the carboxylic acid and to depend on the nature of the alcohol only.

This function $\frac{K_2}{K_2'} M'$, which has been termed the water value of the pure solvent, is identical with r in the expression used by these authors, and is correctly interpreted by them as a hydrolytic constant. Their further conclusion, namely, that the general applicability of the formula is proof of the view that the alcohol forms the reactive complex ion, is, however, clearly incorrect, as the present mathematical investigation shows that the same expression is obtained whether the reactive ion is formed from the alcohol or the carboxylic acid.

The present author has found that this "water value" may be determined for a solvent by studying the effect of water on any of the catalytic changes which have been mentioned, and also on the basis of the tintometric method, referred to later in this paper. Each method, however, gives a slightly different number for the "water value," but this is perhaps inevitable, owing, in part, to the fact that the concentration of catalyst is very different in different experiments, and also that it is at present not easy to determine the influence of the substance undergoing catalytic change. The values are of the same order, however, in each case, and to illustrate what kind of preliminary results have been obtained, the numbers for the "water value" of 50 c.c. of a specimen of nearly absolute alcohol may be given:

Type of catalysis or test.	Esterification.	Acetone bromination.	Hydrazobenzene conversion.	Tinctometric.
Water value	0.22	0.15	0.18	0.12

Variation of the Velocity of Esterification in Alcohol with Alteration in the Amount of Catalyst.

Goldschmidt and Udby (*Zeitsch. physikal. Chem.*, 1907, 60, 735) and Kailan in numerous papers (*Monatsh.*, 1906, 27, 543, 997; *Annalen.*, 1907, 351, 186, etc.) draw attention to the fact that in alcohol containing water, the velocity of esterification at constant volume increases more rapidly than the concentration of the catalyst. The first-named authors consider that this may be explained if the assumption be made that the alcohol forms the complex hydrious to which the reaction velocity is proportional.

In order to ascertain whether this conclusion is a valid one, let one of the complex hydrions in a mixture containing hydrogen chloride be selected, say I'' .

We have $I'' = \frac{H \cdot W''}{K''_2 \cdot V}$, and at constant volume it is obvious that an increased amount of hydrogen chloride could never increase the amount of any of the free bases. K''_2 and V are constant, so that I'' can only increase more rapidly than the concentration of hydrogen chloride if

the same is true of the hydrogen ions. That is, $\frac{d^2H}{dA^2}$ must be positive and $\frac{d^2A}{dH^2}$ negative. •Differentiating the expression connecting the amount of hydrogen chloride with the hydrogen ions, we obtain the expression :

$$\frac{d^2A}{dH^2} = \frac{2}{V} \left[\frac{M}{\sum K_2} + 1 \right] \left[\frac{1}{K_1} + \frac{M}{\sum K K_2} \right],$$

which is always positive, so that the disproportionate increase in the esterification velocity cannot be due to any reaction which is simply dependent on the mass of a complex hydron formed from the alcohol or carboxylic acid.

Of the other individual substances which might be concerned, the relation between the undissociated compound, S'' , the base, W'' , and the hydrogen ions is :

$$S' = \frac{H \cdot W' \cdot R}{K''_2 V} = \frac{H^2 \cdot W'' \cdot \left[\frac{M}{\sum K_2} + 1 \right]}{K''_2 V^2}$$

Any velocity which is proportional to S'' may (providing W'' does not diminish too rapidly with increase in the amount of catalyst, that is, if W'' is a very weak base) increase more rapidly than the amount of catalyst, for

$$\frac{d^2(H^2)}{dA^2} = \left(\frac{dH}{dA} \right)^2 + H \left(\frac{d^2H}{dA^2} \right),$$

which is positive, providing V is less than $2 \left(\frac{1}{K_1} + \frac{M}{K_2 K} \right)$, a number which must be very great. An increase in the velocity of reaction proportionately greater than the increase in catalyst may indicate that an undissociated compound of the catalyst with the changing substance is, in part at least, concerned directly in the formation of the final products.

Neutral Salt Action.

A similar reflection may apply to the acceleration by neutral salts of reactions in which powerful acids in dilute solution take part. The neutral salts must decrease the concentration of the hydrogen ions themselves, and also that of the complex ions of the changing substance, by converting them, on the one hand, into undissociated acid and undissociated compound. It follows from the relations above discussed that when " M " varies but slightly, which is the case if the base is very weak,

$$\frac{S'}{HR} = \frac{H \cdot R}{V} \cdot \frac{M''}{K''_2 K} \cdot \frac{K \cdot V}{H \cdot R} = \frac{K_1}{K''_2 K''} M''$$

is nearly constant, say q .

(18) Hence $dS'' = q.dHR$.

That is, (1) if S'' is large compared with HR , in other words, if the acid is strong, the hydrogen ions and the complex base hydrions on disappearing will form mainly undissociated compounds of the reacting bases; on the other hand, (2) if the acid is weak, then the ions will form mainly undissociated acid.

(1) May account, in part, for the well-known stimulating effect of neutral salts in catalysis by mineral acids, and the disappearance of the hydrogen ions is in accordance with observations, such as that of Walker and Wood (Trans., 1903, 33, 490), that the degree of hydrolysis of the salt of a strong acid with a weak base in aqueous solution, as measured by its hydrolytic power, appears to decrease on addition of a neutral salt of the strong acid.

(2) Accounts for the depressant effect produced by addition of their neutral salts to solutions of weak acid.

Preliminary Determinations by Tinctometric Processes of the Relative Basic Affinities of Common Solvents.

Whilst the majority of modern attempts to explain the phenomena of catalysis by acids have been based on the attribution of a basic character to the reacting compounds, and, although esters have been found to retard esterification and to lower to a slight extent the conductivity of an acid solution (Acree), it is noteworthy that no simple tinctometric method, such as those applied in aqueous solutions to the determination of affinities of acids and bases, has been employed.

Obviously, the experiments would have to be made in solvents with basic affinities of an order not greater than the substances experimented with, so that water, which is a much stronger base than alcohol, could not be used as a medium. It was evident, too, that the indicator required must be a weak base.

The first experiments made were in absolute alcohol. It was found that in this medium a dilute solution of *m*-nitroaniline was suitable for high concentration of mineral acid, and that one of aminoazobenzene was better for small concentrations, $N/100$ or less. The red colour of the acidified aminoazobenzene solution was partly discharged on the addition of minute quantities of water, and with the quantity of acid required to restore the colour to the original amount, was nearly proportional to the amount of water present if the alcohol itself was assigned a small water value (50 c.c. of alcohol was equivalent to 0.12 c.c. of water), so that hydrolysis of aminoazobenzene hydrochloride in alcohol much resembled esterification in alcohol, which serves experimentally to connect the two phenomena by experiment.

The results in ether, ethyl acetate, ethyl formate, and methyl alcohol

ere of the same character, except that these, in accordance with their different basic affinities, as seen below, had different water values.

Apart from the fact that benzene, light petroleum, and carbon tetrachloride do not dissolve water and are generally less useful as solvents, these liquids, as might be expected from their more nearly neutral character, appear more suitable solvents for determination of the relative affinities of very weak bases.

The author is much indebted to Mr. R. W. L. Clarke for conducting an independent series of measurements of the relative basic affinities of a number of common bases in several solvents. He reports as follows:

The results under the head *A* were carried out in 99.65 per cent. alcohol with hydrogen chloride about 0.025*N* (with aminoazobenzene about 1 in 500,000 of solvent). Known quantities of the bases were added to the same amount of solution, and then the quantity of acid alcoholic hydrogen chloride required to impart the same tint to each was introduced.

Under *B* are given results in benzene with trichloroacetic acid, and under *C* with carbon tetrachloride and trichloroacetic acid, used in 5 per cent. solution as a maximum concentration. The relative basic affinities were assumed to be proportional to the amount of acid used at constant total volume. The numbers refer to equimolecular proportions, and the basic affinity of water is taken as 1, but that of ethyl acetate is kept constant to link the three series together.

Base.	<i>A.</i>	<i>B.</i>	<i>C.</i>
Carbamide.....	17.5	—	—
Water	1.0	—	—
Ether.....	—	0.13	0.12
Ethyl alcohol	—	0.06	0.06
Acetone.....	—	0.055	0.045
Ethyl acetate	0.05	0.05	0.05
* Methyl alcohol.....	0.015	0.02	0.01

The bases used were highly purified materials, and small quantities of impurities might conceivably have made an appreciable difference in the series *A*, but could not very greatly affect *B* and *C*, where the molecular proportion of trichloroacetic acid was considerable.

Specimens of ether, some dried by distillation over sodium and others over phosphoric oxide, gave concordant results.

* It is remarkable that, although methyl alcohol in these solvents appears to act as a specially weak base, yet in the liquid state (that is, when used as a solvent) it has a water value much greater than ethyl alcohol, whether this be estimated tintometrically or otherwise (compare Goldschmidt and Udby, *loc. cit.*, p. 755). This, among many other points, requires careful investigation.

Part II.

An Explanation of the Properties of Acids not Necessarily Involving the Conception of Hydrogen Ions.

Proceeding from the usual standpoint that the catalytic activity and the salt-forming powers of acids are dependent on the hydrogen ions, it has been shown in the preceding pages that the principal properties of acid in aqueous and similar solutions may be fully explained, no matter what may be the actual concentration of the hydrogen ions in any given case. From a mathematical point of view, this must also be true if their concentration is always $=0$, and would lead to the conclusion that the properties of acids when dissolved in solvents containing bases may merely depend on (1) the extent to which they combine with the bases present, (2) the manner in which they are partitioned between the bases, and (3) the degree to which the resulting salts are dissociated.

The condition that the hydrogen ions are absent in the equation discussed is that $K_1=0$, or unreal; it follows that $\frac{1}{K_2}$, $\frac{1}{K_2}$, etc. are unreal, but that the products $\frac{K_1}{K_2}$, $\frac{K_1}{K_2}$, etc. are real. Call these ϕ , ϕ' , etc.; they represent the mutual *propensities** of the bases for the acid. K_1 , $\frac{1}{K_2}$, etc., are here unreal, because they are not exhibited until the base and the acid are brought into contact.

As there is no necessity for supposing that the hydrogen ions exist in any but infinitesimal concentration, it would appear more satisfactory to revise the prevailing views of the nature of acids and bases, employing a convention of the above character, as this admits of an advance from a practical point of view. At present the function of an acid which is regarded as the measure of its affinity is its dissociation constant in water; this, however, depends on two different real functions, namely, the affinity of the acid for the water and the dissociation of the hydroxonium salt. It should not be too difficult to separate these by experimental treatment.

The new relations which arise by eliminating the incommensurable functions in the preceding formula, and which appear capable of practical application, may be tabulated here, and will be applicable whether the hydrogen ions are real or not.

$$(19) \quad x = \sum I = R, \quad I'' = \frac{RM''\phi''}{\sum M\phi}, \text{ etc.},$$

* The term "affinity" has been used in other senses, and the function it is here proposed to term "propensity" refers to the tendency of the acid and the base to yield a compound which is a perfect electrolyte.

where x is the degree of dissociation of the acid as at present measured, ΣI the sum of the positive base ions, I'' represents the amount of any one of these, and R the gram-equivalents of the negative ions whether united with solvent or not.

$$(20) \quad k = \frac{\Sigma M\phi}{\Sigma \frac{M\phi}{K} + 1},$$

where k is the dissociation constant as generally understood, and K is the dissociation constant of the hydroxonium salts.

$$(21) \quad x^2 + \frac{\Sigma M\phi}{\Sigma \frac{M\phi}{K} + 1} Vx = \frac{\Sigma M\phi}{\Sigma \frac{M\phi}{K} + 1} V,$$

where V is the volume in c.c. containing one gram-equivalent of the acid.

$$(22) \quad HR = \frac{x^2}{V \Sigma M\phi},$$

where HR is the uncombined acid

$$(22a) \quad \Sigma S = \frac{x^2 \Sigma \frac{M\phi}{K}}{V \Sigma M\phi} \quad S'' = \frac{x^2 M'' \phi''}{K'' V \Sigma M\phi},$$

where S is the sum of the amounts of all the undissociated salts and compounds of the acid with the bases, and where S'' is the amount of these, derived from any one base.

In dealing with an aqueous solution, the sign Σ is, of course, omitted if no other base than water is present.

The functions x and k are already known for many important acids. It should be possible to determine the amounts of the free acids and the hydrated acids in their aqueous solutions at known concentration. There will then be an experimental basis for the determination of the relative potencies, apart from the basic power of the solvent. Probably, however, it will be much simpler to deal with the problem first in non-ionising solvents, but in this case the undissociated or non-conducting compounds of acid and base will probably be of the most importance.

Dissociation Constants of Acids.

An interesting relation may be pointed out for the case of acids. Here in aqueous solution :

$$(23) \quad k = \frac{KM\phi}{M\phi + K} \text{ or } M\phi = k \frac{K}{K - k},$$

where K is the dissociation constant of the hydroxonium salt. If, as appears probable, these salts are largely dissociated in the same way

as ammonium salts, then k may be neglected in the denominator in the last equation if the acid is very weak; and

$$(24) \quad M\phi = k,$$

that is, for a weak acid, if its molecule is not hydrated in aqueous solution, its dissociation constant is identical with its propensity for liquid water.

Application to the Case of the Hydrolysis of the Salt of a Weak Base.

As measured by the catalytic activity of the solution of the hydrochloride of a base, say carbamide, the connexion may be arrived at as follows:

The water may be represented as W , the base, W' , and the hydrolyte, W'' . The velocity of hydrolysis of W'' is mainly dependent on its capacity to form the ions I'' . This is given by $I'' = \frac{RM''\phi''}{\Sigma M\phi}$, that is,

proportional to $\frac{R}{\Sigma M\phi}$ (or when the substance catalysed is a very weak base and does not appreciably affect the state of the solutions) or proportional to $\frac{R}{M\phi + M'\phi'}$, where M and ϕ refer to water, and M' and ϕ' to the base W' .

R in the case of dilute hydrochloric acid is nearly constant whether a base like carbamide is present or not.

Let 1 gram-molecule of the hydrochloride be placed in water, the apparent degree of dissociation, X , into base and free acid is:

$$\frac{I'' \text{ where carbamide is present}}{I'' \text{ where carbamide is absent}} = \frac{R}{M\phi + M'\phi'} \cdot \frac{M\phi}{R} = \frac{M\phi}{M\phi + M'\phi'}.$$

$$\text{As } M' = \frac{\text{total amount of free urea}}{\text{volume}} = \frac{X}{V}, \text{ and as } M \text{ for dilute aqueous}$$

$$\text{solution remains nearly constant, } X = \frac{M\phi}{M\phi + \frac{X}{V}\phi'}, \text{ or } \frac{X^2}{V\phi'} + M\phi X = M\phi.$$

$$(25) \text{ hence } X^2 + \frac{M\phi}{\phi'} VX = \frac{M\phi}{\phi'} V, \text{ or } X^2 + kVX = kV.$$

This is identical with the formula of Arrhenius (*Zeitsch. physikal. Chem.*, 1890, 5, 16), and was found by Walker and Wood (Trans., 1903, 33, 489) to apply to the case of carbamide hydrochloride, the dissociation constant k being $= \frac{M\phi}{\phi'}$ in the new measures.

Walker and Wood find $k = 0.781$ at 25° when the volume is expressed in litres. Putting M , the active mass of liquid water, as $\frac{1000}{18}$, $\frac{\phi'}{\phi}$ becomes $\frac{1000}{18 \times 0.781} = 71.1$. That is, for hydrochloric acid, the

propensity of dissolved carbamide is 71.1 times the propensity of liquid water.

Comparing this with the value $\frac{\phi'}{\phi}$ in alcoholic solution, it appears that liquid water has only one-fourth the basic affinity of water dissolved in alcohol.

The relations given in Part II of this paper will be true in practice whether the reality or otherwise of free hydrogen ions is assumed.

In conclusion, it must again be pointed out that the conceptions introduced in this and the preceding paper (this vol., p. 2163) have, for the most part, been used before. In particular, the view that acids, unlike certain metallic hydroxides and salts, are not of themselves electrolytes, is suggested in Lothar Meyer's "Modern Theories of Chemistry" (Bedson and Walker's translation, 1888 edition, p. 535), and attributed to Thomsen (1874). Lowry has recently drawn attention to the great importance of the point.

The only suggestion regarded as in any sense a novel one, is that water, when added to acids in less basic solvents, reduces the concentration of the hydrogen ions, or, on a less hypothetical basis, diminishes the availability of the acid for salt formation. Even this appears to be in a measure anticipated by Armstrong's proposition that substances which act as dehydrants will have a "concentrating" effect on others which are hydrated in aqueous solution.

The author desires to express his indebtedness to the Government Grant Committee of the Royal Society for a grant, which defrayed much of the cost of the experiments.

CCXXII.—*The Oxidation of Phosphorous Acid by Iodine.*

By BERTRAM DILLON STEELE.

In a former paper (Trans., 1907, 91, 1642), it has been shown that the velocity of the reaction between iodine and hypophosphorous acid in acid solution is independent of the concentration of the former, and proportional to that of the latter, reagent, and that the reaction is catalytically accelerated by hydrogen ions, no appreciable action occurring in their absence.

In the same communication, it was pointed out that the oxidation of phosphorous acid by iodine appeared to be retarded by hydrogen ions, and that this remarkable difference in behaviour had been utilised by

Rupp and Finck (*Arch. Pharm.*, 1902, **240**, 663) in developing a method for the estimation of phosphorous and hypophosphorous acids either alone or when mixed.

The present communication describes the results that have been obtained during the attempt to elucidate the mechanism of the reaction between iodine and phosphorous acid under different conditions.

Federlin (*Zeitsch. physikal. Chem.*, 1902, **41**, 565), while investigating the reaction between iodine, phosphorous acid, and potassium persulphate, carried out a few experiments in which he measured the rate of reduction of iodine by a large excess of phosphorous acid, and he concluded that this was a bimolecular reaction.

His experiments may, indeed, be interpreted as showing that the reaction is approximately unimolecular with respect to the iodine, but they give no information as to the number of reacting molecules of phosphorous acid. These experiments were subsidiary, and were carried out by Federlin only to enable him to interpret the results of his other experiments. He does not appear to have noticed the extraordinary effect of reducing the acid concentration.

Iodine and phosphorous acid appear to be capable of undergoing two distinct reactions, of which one predominates in acid solution, and the other in the absence of any appreciable concentration of hydrogen ions.

The mechanism of the former of these reactions has been worked out with tolerable certainty, and the conclusions which have been arrived at are described in the sequel, but it has not been found possible to suggest a hypothesis which is competent to explain the mechanism of the second reaction.

The phosphorous acid for the experiments was prepared by the action of distilled water on phosphorus trichloride which had been previously purified by fractional distillation. The resulting solution containing a mixture of phosphorous and hydrochloric acids was then evaporated in a vacuum, diluted with water, and again evaporated, this process being repeated until the solution was quite free from hydrochloric acid. It was finally diluted so as to obtain a solution containing half a gram-molecule per litre.

The reaction was carried out in a thermostat at 35°, at which temperature the iodine completely disappeared in from two to eight hours according to the concentration of the mixture. The reagents were immersed separately in the thermostat, and mixed after they had attained the temperature of the bath. Samples of the mixture were then withdrawn from time to time, and the residual iodine was titrated with a fiftieth molar solution of sodium thiosulphate.

On account of the practical impossibility of estimating the concentration of the phosphorous ions during the experiments, the phosphorous acid was always present in considerable excess. In these circumstances, the degree of ionisation of the acid remained practically constant throughout each experiment, and the concentration of the reacting substance, whether ion or molecule, might without appreciable error be taken as proportional to the total concentration.

The Influence of the Iodine Concentration.—In order to determine the influence of the iodine concentration, a number of experiments was carried out, in which a constant concentration of phosphorous acid was allowed to react with varying concentration of iodine, the acid being always in excess.

In the equation :

$$\frac{dx}{dt} = k(A-x)^m(B-x)^n,$$

in which A represents the initial concentration of the phosphorous acid and B that of the iodine, it was anticipated that Federlin's conclusion that $n=1$ would be confirmed; it was found instead that $n=0.5$. On the assumption that $(A-x)$ is constant and that $n=1$, integration of the foregoing equation gives the usual equation for a unimolecular reaction :

$$k(A-x)^m = K = \frac{1}{t} \log \frac{B}{B-x} \quad (1);$$

if, however, $n=0.5$, we get the equation :

$$\frac{dx}{dt} = k(A-x)^m(B-x)^{0.5},$$

and on integration :

$$k(A-x)^m = K = \frac{2}{t} \left(\sqrt{B} - \sqrt{B-x} \right) \quad (2).$$

Table I shows a typical example of the manner in which the experimental results are described by equations (1) and (2) respectively.

TABLE I.

t .	$B-x$.	K for $(n=1)$.	K for $(n=0.5)$.
0	0.0190	—	—
67	0.0154	0.00314	0.00206
113	0.0132	0.00324	0.00203
177	0.0104	0.00340	0.00203
232	0.0083	0.00356	0.00201
265	0.0075	0.00350	0.00193

t = time in minutes at which titration was made. $(B-x)$ = iodine concentration in milligram-mols. per c.c. at the time t . The values of the constants calculated from equations (1) and (2) are given in the third and fourth columns.

Experiments 2 to 18, which are given later and which take into account the variation, not only of the iodine, but also of the phosphorous acid concentration, confirm the assumption that the velocity of the reaction is proportional to the square root of the iodine concentration.

The Influence of Phosphorous Acid Concentration.

The evaluation of the number of reacting molecules of phosphorous acid is complicated by the fact that the reaction is catalytically accelerated by the presence of hydrogen ions, which are not only produced from the phosphorous acid, but are also increased in quantity during the reaction. The reaction, in all probability, undergoes autocatalytic acceleration in the same manner as was found to be the case in the oxidation of hypophosphorous acid by iodine, but partly on account of the extreme slowness of the reaction when small concentrations of phosphorous acid were used; no experiments have been carried out in which the extra quantity of hydrogen ions produced would exert any appreciable effect on the velocity.

The influence of the phosphorous acid was investigated by varying its concentration in different experiments. These experiments showed that the reaction is unimolecular with respect to the phosphorous acid, so that the complete equation for the velocity is as follows:

$$\frac{dx}{dt} = k(A-x)(B-x)^{\frac{1}{2}} + k'C(A-x)(B-x)^{\frac{1}{2}} \quad \dots (3).$$

This equation involves the following hypotheses: The velocity is proportional to the concentration of the phosphorous acid and to the square root of the iodine concentration; it is catalytically accelerated by hydrogen ions, the concentration of which is represented by C .

The velocity coefficient, k , of the now catalysed reaction may usually be put equal to zero, since in most cases it is extremely small compared with k' , the coefficient of the catalysed reaction.

In the present case, this cannot be done, as k and k' are comparable in magnitude.

The hydrogen ion concentration, C , is made up of the sum of H^+ produced by the phosphorous, phosphoric, hydriodic, and added acids, and cannot be evaluated with any approach to exactness. This is due in the first place to the uncertainty which attaches to the ionisation coefficient of a polybasic acid, and, secondly, to the unknown influence of each acid on the dissociation of the others. Strictly speaking, $C = [a(A-x) + a'x + a''c + a'''2x]$, where $A-x$, x , $2x$, and c are the concentrations of the phosphorous, phosphoric, hydriodic, and added acids respectively, and a , a' , a'' , and a''' are the corresponding ionisation coefficients of each acid in the presence of all the others.

On account of the impossibility of accurately evaluating these coefficients, the following simplification has been made:

a , a' , and a'' are of the same order of magnitude, a'' being greater than a , and a greater than a' .

In the absence of added acid, the expression for the hydrogen ion concentration becomes $aA + (a' + 2a'' - a)x$, which, on account of the small magnitude of x compared with A , may without sensible error be put equal to aA .

That no appreciable error has been introduced by this assumption will be evident from a consideration of experiments 1 to 9.

The accurate evaluation of the hydrogen ion concentration is rendered impossible in the case of the experiments where sulphuric acid has been added by our ignorance of the mutual influence of strong acids on the ionisation coefficients.

Notwithstanding this, the effect of increasing the hydrogen ion concentration will be clearly seen by reference to the results of experiments 10 to 15.

If equation (3) is written in the form

$$\frac{dx}{dt} = (k + k'C)(A - x)\sqrt{B - x},$$

we obtain on integration the equation:

$$(k + k'C) = \frac{2}{t\sqrt{A - B}} \left(\tan^{-1} \sqrt{\frac{B}{A - B}} - \tan^{-1} \sqrt{\frac{B - x}{A - B}} \right). \quad (4)$$

and the constant obtained by the use of this equation is given in the last column of the following table:

TABLE II.

No. of expt.	A.	B.	C.	Duration of expt., in minutes.	$k + k'C$.		
					Lowest.	Highest.	Mean.
1	0.2	0.0093	0.0866	401	0.00193	0.00225	0.00212
2	0.2	0.019	0.0866	266	0.00200	0.00208	0.00205
3	0.2	0.0285	0.0866	340	0.00181	0.00191	0.00186
4	0.2	0.0382	0.0866	402	0.00152	0.00173	0.00169
5	0.3	0.00976	0.12	167	0.00249	0.00249	0.00249
6	0.3	0.01936	0.12	240	0.00242	0.00275	0.00268
7	0.3	0.0286	0.12	246	0.00221	0.00254	0.00250
8	0.4	0.0096	0.143	111	0.00263	0.00240	0.00230
9	0.4	0.0192	0.143	196	0.00260	0.00304	0.00287
						0.00294	0.00285

The general correctness of the hypotheses contained in equation (3) is indicated by the very slight variation of the constant in any single experiment. Its insufficiency is shown in table III by the variation of $k + k'C$ in experiments 1, 2, 3, and 4, all of which have the same A and C values, and similarly in experiments 5, 6, and 7 and 8 and 9. In all these cases, but most markedly in those which have the

smallest A and C values, there is a decrease in $k+k'C$ with increasing iodine concentration.

To ascertain in what direction it would be necessary to modify equation (3) in order that it may more accurately describe the experimental results, k and k' must first be evaluated. This can easily be done, since we have in table IV nine equations giving values of $k+k'C$, and C is known, at least approximately, for all of them.

TABLE III.

Expt.	A .	B .	C .	$k+k'C \times 10^3$.	$k \times 10^3$.	$k' \times 10^3$.
1	0.2	0.01	0.087	2.12	0.88	14.3
2	0.2	0.02	0.087	2.05	0.90	13.2
3	0.2	0.03	0.087	1.86	0.71	13.2
4	0.2	0.04	0.087	1.69	0.54	13.2
5	0.3	0.01	0.12	2.68	0.92	14.8
6	0.3	0.02	0.12	2.50	0.94	13.0
7	0.3	0.03	0.12	2.30	0.72	13.2
8	0.4	0.01	0.15	2.87	0.96	12.7
9	0.4	0.02	0.15	2.85	0.94	12.7

TABLE IV.

$k \times 10^3$ calculated from experiments.							Mean
Expt.	1 and 5.	1 and 8.	5 and 8.	2 and 6.	2 and 9.	6 and 9.	3 and 7.
1	16.5	12.2	—	—	—	—	—
5	16.5	—	13.2	—	—	—	—
8	—	12.2	—	—	—	—	—
2	—	—	—	13.5	13.0	13.1	—
6	—	—	—	13.5	—	12.5	—
9	—	—	—	—	13.0	12.5	—
3	—	—	—	—	—	—	13.2
7	—	—	—	—	—	—	13.2
General mean							13.4

k and k' were calculated from different pairs of experiments, those experiments being selected which had the same value for the iodine concentration B . The values of k' calculated in this manner from seven pairs of experiments are given in table IV. In only one calculation, that from experiments 1 and 5, does the value differ to any extent from the general mean of 13.4×10^{-3} . This exception appears to be due to an abnormal value for the constant from experiment 5. The values of k which are given in the sixth column of table III were obtained by deducting the numbers in column 7 from those in column 5. This method of calculation throws practically all the variation in $k+k'C$ on k , the non-catalytic constant, and this conclusion is confirmed by evaluating k by the method employed for k' and described in table IV; such a calculation yields values for k which, like those in column 6 of table III, diminish with increasing concentration of iodine, and this suggests that it is the

1st term in equation (3) which is inaccurate. On the whole, except for this slight variation of k , the experimental results are well expressed by the equation used.

The Influence of Added Acid.

In the series of experiments 10 to 15 (table V), sulphuric acid has been added in varying quantity, the concentration in each experiment being given under c . In all cases an increase in c produces an increase in the magnitude of the constant $k+k'C$, but although, as in the former series, the constant is very good in each experiment, no simple relation between its values from different experiments can be detected. This, as already pointed out, is due to the uncertainty as to the total H^+ concentration.

TABLE V.

No. of expt.	A.	B.	c.	$C(=aA+a''c)$	Duration of expt., in minutes.	$k+k'C$		
						Lowest.	Highest.	Mean.
10	0.2	0.019	0.02	0.1146	272	0.00220	0.00226	0.00222
11	0.2	0.0187	0.05	0.1146	280	0.00246	0.00265	0.00254
12	0.2	0.019	0.10	0.203	311	0.00278	0.00304	0.00296
13	0.2	0.0193	0.4	0.447	229	0.00408	0.00468	0.00444
14	0.3	0.0194	0.05	0.181	235	0.00284	0.00305	0.00299
15	0.3	0.0194	0.10	0.236	227	0.00331	0.00369	0.00353

On the assumption that this is given approximately by $(aA+a''c)$, the sum of the concentrations of the hydrogen ions from the phosphorous acid and from the sulphuric acid, each in the absence of the other, values are obtained for k' which vary between 9.7 and 6.7.

The calculation may be reversed, and the total hydrogen ion concentration calculated from the values of k and k' found already from experiments 1 to 9. This has been done, and table VI contains, for experiments 10 to 15, the values of the concentrations of the phosphorous acid (A), iodine (B), and sulphuric acid, c , as well as that of the summed constants ($k+k'C$), the last two columns showing the difference between $(aA+a''c)$ and C calculated in this manner. The

TABLE VI.

Expt.	A.	B.	c.	$k+k'C$	$aA+a''c$	C
10	0.2	0.02	0.02	2.22	0.1146	0.1
11	0.2	0.02	0.05	2.54	0.143	0.123
12	0.2	0.02	0.10	2.96	0.203	0.157
13	0.2	0.02	0.4 [HCl]	4.44	0.427	0.271
14	0.3	0.02	0.05	2.84	0.181	0.16
15	0.3	0.02	0.10	3.53	0.236	0.201

after is always less than the former, and by an amount which might be expected from the decrease in ionisation of the phosphorous acid, due to the presence of the sulphuric acid.

This decrease in ionisation would have a dual effect if the reaction were one in which phosphorous ions are oxidised by iodine, since the concentration of the phosphorous ions would also be diminished, and hence also the observed constant.

The fact that experiment 13 gives for C a value (0.271) which is less than the H^+ concentration from the added acid alone, is the only one in the whole of the present investigation which indicates in any way whether the iodine reacts with ion or undissociated molecule.

The Reaction with Very Low Hydrogen Ion Concentration.

It has been already mentioned that if the H^+ concentration is made very small by the addition of a salt of a very weak acid, such as the sodium salts of acetic, carbonic, or boric acid, the reaction velocity is increased enormously. With moderate concentrations of iodine and excess of phosphorous acid, reaction is complete in from one to seven minutes at 25° . In order to make any measurements, it is therefore necessary to work at moderately low temperatures, and the following experiments, in which a large excess of phosphorous acid was always used, were carried out in a bath of melting ice.

Experiment 18, table VII, will show the nature of the results which have been obtained.

TABLE VII.—Experiment 18.

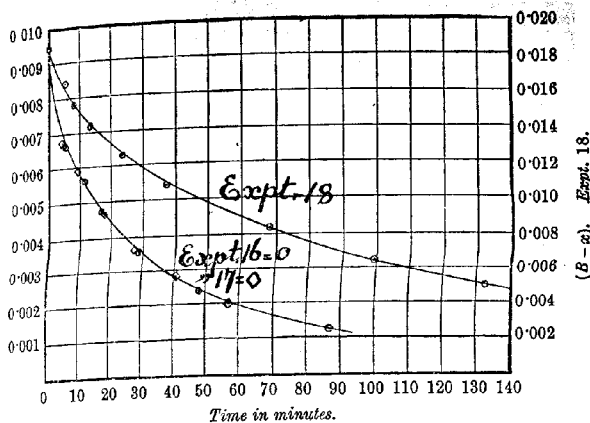
$A=0.2$. $B=0.02$. $C_2H_3O_2Na=0.4$. $KI=0.050$.

t	$B-x$	$1/\log \frac{B}{B-x}$	$1/tB \frac{x}{B-x}$
0.0	0.0189	—	—
5.5	0.0169	0.0209	1.165
8.5	0.0155	0.0238	1.37
13.5	0.0144	0.0201	1.22
23.5	0.0125	0.0175	1.15
37.5	0.0108	0.0156	1.05
65.5	0.0084	0.0119	0.98
99.5	0.00644	0.0108	1.02
142.5	0.0050	0.0093	1.04
230.0	0.00328	0.0077	1.10

Although this and the other experiments of this series yield approximately constant values for the bimolecular expression $\frac{1}{tB} \frac{x}{B-x}$ (column 4, table VII), the conclusion that the reaction is bimolecular with respect to the iodine is not confirmed by the consideration of the value of the constant from different experiments. The relation between the reaction velocity and the concentration of the reagents is, indeed, so complex that the attempt to give quantitative expression to the results has been abandoned.

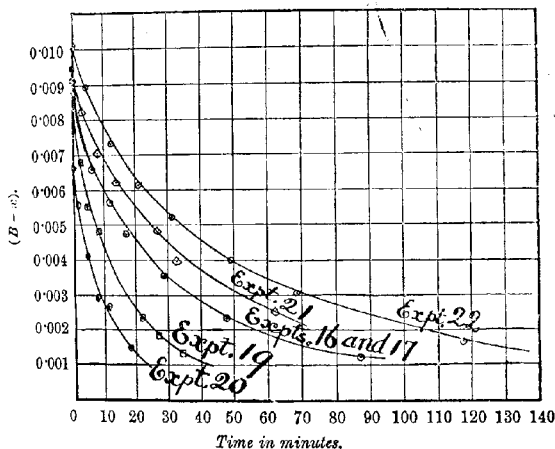
The curves of Figs. 1 and 2 are obtained by plotting the experimental results which are given in table VIII. This table contains the

FIG. 1.



times of measurement expressed in minutes, and the corresponding values of the iodine concentration for experiments 16 to 22, the con-

FIG. 2.



centrations of the phosphorous acid (A), iodine (B), sodium acetate, and potassium iodide being given for each experiment.

TABLE VIII.—Experiment 16.

 $A=0.2$. $B=0.01$. $C_2H_3O_2Na=0.4$. $KI=0.025$.

t	0.0	5.5	11.5	17.0	29.0	48.0	87.0
$(B-x) \times 10^3$...	9.44	6.60	5.66	4.76	3.54	2.36	1.2

*Experiment 17 repeats Experiment 16.**Experiment 18.* $A=0.2$. $B=0.02$. $C_2H_3O_2Na=0.4$. $KI=0.050$.

t	0.0	5.5	8.5	13.5	23.5	37.5	68.5	99.5	142.5
$(B-x) \times 10^3$...	18.9	16.8	15.6	14.4	12.5	10.8	8.4	6.4	5.0

Experiment 19. $A=0.2$. $B=0.01$. $C_2H_3O_2Na=0.6$. $KI=0.025$.

t	0.0	2.5	4.5	8.0	22.0	27.0	34.0
$(B-x) \times 10^3$...	8.84	6.82	5.54	4.80	2.38	1.86	1.36

Experiment 20. $A=0.2$. $B=0.01$. $C_2H_3O_2Na=0.8$. $KI=0.025$.

t	0.0	1.5	4.0	7.5	11.25	18.5
$(B-x) \times 10^3$	6.70	5.54	4.12	2.94	2.70	1.50

Experiment 21. $A=0.2$. $B=0.01$. $C_2H_3O_2Na=0.4$. $KI=0.125$.

t	0.0	3.0	8.0	14.0	27.0	33.0	62.0
$(B-x) \times 10^3$...	9.14	8.20	7.08	6.22	4.82	3.98	2.54

Experiment 22. $A=0.2$. $B=0.01$. $C_2H_3O_2Na=0.4$. $KI=0.225$.

t	0.0	4.0	12.5	21.0	31.5	49.5	68.5	112.5
$(B-x) \times 10^3$...	10.06	8.92	7.32	6.16	5.20	4.00	3.08	1.68

The same experiments are summarised in table IX, which gives, in

TABLE IX.

Expt.	A .	B .	$C_2H_3O_2Na$.	KI .	t .
16 and 17...	0.2	0.01	0.4	0.025	20
18.....	0.2	0.02	0.4	0.05	53
16 and 17...	0.2	0.01	0.4	0.025	20
19.....	0.2	0.01	0.6	0.025	9.5
20.....	0.2	0.01	0.8	0.025	7
16 and 17...	0.2	0.01	0.4	0.025	20
21.....	0.2	0.01	0.4	0.125	29
22.....	0.2	0.01	0.4	0.225	34

addition to the data contained in table VIII, the time required in the case of each experiment for the reaction to proceed half way to com-

lation; these figures are given in the sixth column. The great complexity of the reaction and its extreme sensitiveness to concentration changes will be clearly seen from a study of this table and of the figures.

Thus experiments 16, 17, and 18, which are shown in Fig. 1, show the effect of doubling the iodine concentration, the time of half reaction being increased from twenty to fifty-three minutes, an effect which is exactly the opposite to that which should occur in a bimolecular reaction.

Experiments 16, 17, 19, and 20 (Fig. 2) show the acceleration of a reaction velocity by an increase in the sodium acetate concentration. This corresponds with a diminution in the concentration of the hydrogen ions, and, if this conditions an increase in the velocity, it is obvious that the reaction in the conditions under discussion cannot be the same as that which takes place in the presence of much free acid.

Finally, the effect of adding potassium iodide (or iodine ions) is seen from experiments 16, 17, 21, and 22 to be a retardation, the time of half reaction being increased from 20 in experiments 16—17, 29 and 34 respectively in experiments 21 and 22.

Summary.

It has been shown that two distinct reactions occur between iodine and phosphorous acid, one of which preponderates in acid solution and the other in the absence of any strong acid.

The former of these reactions is catalytically accelerated by the presence of hydrogen ions, and the reaction velocity is proportional to the concentrations of the phosphorous acid (or of the phosphorous ion) and to the square root of the concentration of the iodine.

The conclusion is drawn that reaction takes place between phosphorous acid molecules (or ions) and iodine atoms, the latter being supplied in extremely small, but sufficient, quantity by the dissociation of the iodine molecules.

A slight apparent retardation of the reaction velocity by the iodine is probably due to the incompleteness of the hypothesis which has been suggested to explain the experimental results.

The second reaction, which takes place in the presence of salts, such as sodium acetate, bicarbonate, and borate, is extremely complex; the reaction being accelerated by the presence of sodium acetate, retarded by the presence of iodine, and also retarded by the presence of iodide ions.

No hypothesis explanatory of this reaction has been suggested.

OBITUARY NOTICES.

WILLIAM HENRY PERKIN.*

BORN MARCH 12TH, 1838; DIED JULY 14TH, 1907.

SIR WILLIAM HENRY PERKIN, whose death occurred on July 14th, 1907, was born in London on March 12th, 1838. He was the youngest son of Mr. George Fowler Perkin, a builder and contractor, who died in 1865 at the age of 63. The younger Perkin received his early education at a private school, and was afterwards sent to the City of London School, where it may be said that his inborn talent for chemistry as a science first took definite form through the encouragement of the late Thomas Hall, who was at that time one of the class masters in the school. Science at that period apparently did not form a recognised part of the educational curriculum, since Mr. Hall had to take the time for giving two weekly lectures on chemistry and natural philosophy out of the dinner interval. The schoolboy Perkin attended these lectures with the greatest delight, often sacrificing the midday meal in his enthusiasm, and was soon promoted to the, to him, proud position of being allowed to prepare the experiments, and help Mr. Hall with the demonstrations during the lectures.

It is evident that in the case of Perkin, as is so generally the case with those who leave their mark upon any branch of science, the particular specialisation of faculty and disposition indicative of inherent ability revealed itself at a comparatively early age, and it is certainly a fortunate circumstance that at this critical period of his career he should have fallen under the influence of Mr. Hall, who was himself a pupil of Hofmann's, and who, according to all accounts furnished by contemporaries, must have been highly inspiring as a teacher of science. Perkin has quite recently placed upon record the history of his early life in the following passage:—

"As long as I can remember, the kind of pursuit I should follow during my life was a subject that occupied my thoughts very much. My father being a builder, the first idea was that I

* This notice has been compiled from those previously published by the Royal Society and the Society of Dyers and Colourists.

ould follow in his footsteps, and I used to watch the carpenters at work, and also tried my hand at carpentering myself. Other things I noticed led me to take an interest in mechanics and engineering, and I used to pore over an old book called 'The Artisan,' which referred to these subjects and also described some of the steam engines then in use, and I tried to make an engine myself and got as far as making the patterns for casting, but I was unable to go any further for want of appliances. I had always been fond of drawing, and sometimes copied plans for my father, whose ambition was that I might be an architect. This led me on to painting, and made me think I should like to be an artist, and I worked away at oil-painting for some time. All these subjects I pursued earnestly and not as amusements, and the information I obtained, though very elementary, was of much value to me afterwards. But when I was between twelve and thirteen years of age, a young friend showed me some chemical experiments, and the wonderful power of substances to crystallise in definite forms, and the latter especially struck me very much, with the result that I saw there was in chemistry something far beyond the other pursuits with which I had previously been occupied. The possibility also of making new discoveries impressed me very much. My choice was fixed, and I determined if possible to become a chemist, and I immediately commenced to accumulate bottles of chemicals and make experiments."

It was at this period that Perkin entered the City of London School, and, as he has told us in the passage just quoted, with a distinct bias towards chemistry as a career. This decision appears to have caused his father some disappointment, as at that time chemistry as a profession offered but few attractions, and it was only through the intercession of Mr. Hall that he was allowed, at the age of fifteen, to enter the Royal College of Chemistry as a student under Hofmann in the year 1853. His special ability must have revealed itself also to the eminent professor who was at the head of that institution, for he soon passed through the ordinary course of training, consisting of qualitative and quantitative analysis and gas analysis, and, by the end of his second year, and, under Hofmann's guidance, carried out his first piece of research work. In describing this period of his career in a speech delivered in New York in October, 1906, Perkin significantly added with respect to the ordinary curriculum which all students of the Royal College of Chemistry went through at that time:—"This looked upon only as a preliminary part of my chemical acquirements and not, as many used to and some still do, as a full equipment. Research was my ambition. . . ."

For a youth with these proclivities, no more inspiring influence existed in this country than that exercised by Hofmann in the research laboratory in Oxford Street, and at the age of seventeen we find Perkin, who had by then proved his capabilities, enrolled as honorary assistant to the Professor. In that laboratory the first serious insight into research methods was acquired, and it is of particular interest to note that his initiatory work, instigated by Hofmann, was in connexion with the hydrocarbon anthracene, a substance which, a few years later, served as the starting point in one of the most brilliant synthetical achievements in scientific and industrial chemistry, with which the name of Perkin will be always associated. No less interesting is the circumstance that this first research, although, for reasons which are now readily intelligible, ending in negative results, in no way daunted the ardour of the young investigator, who, in later life, frequently declared that his first efforts at getting definite products from anthracene were of invaluable service to him when he again took up the study of this hydrocarbon from the scientific and technical point of view. The problem set by Hofmann was, in fact, not solved until more than a quarter of a century after Perkin's first attempt, and then by a very indirect method. The general subject which, among others, was under investigation in the Oxford Street laboratory at that time was the production of organic bases from hydrocarbons by the reduction of the nitro-derivatives. Anthracene, then known as "paranaphthalene," had not been brought within the range of these experiments, and the task of isolating the hydrocarbon from coal-tar pitch with the view of nitrating the pure substance was entrusted to Perkin, whose difficulties in attempting on a laboratory scale to achieve a result which is only satisfactorily accomplished on a factory scale are readily imaginable. However, the aid of the tar distiller was invoked, and a supply of the raw anthracene obtained from the Bethels Tar Works, but the pure hydrocarbon could not be nitrated, and so the desired amine corresponding with aniline could not be obtained. As a matter of fact, Perkin had unwittingly produced, by the action of nitric acid on anthracene, the parent substance of alizarin, anthraquinone, although his analyses failed to reveal the nature of the compound, because at that time an erroneous formula had been assigned to the hydrocarbon by its discoverers, Dumas and Laurent. Other (haloid) derivatives of anthracene prepared during the research for a similar reason failed to give intelligible results on analysis, and the young investigator was therefore given another piece of work, namely, the study of the action of cyanogen chloride on naphthylamine, this being a part of a general research on the

ction of cyanogen chloride, etc., upon organic bases, which had, or some time, been going on under the auspices of Hofmann. This second investigation was brought to a successful issue and communicated a year later to the Chemical Society of London, which then held its meetings at a house in Cavendish Square.

Perkin's first successful research was thus completed in 1855 and appeared in the *Journal of the Chemical Society* in 1856 (9, 8; also *Annalen*, 98, 238), from which time, throughout the whole period of his career, this Society received and published practically the whole results of his scientific labours.

The compound described by Perkin in his first paper as "menaphthylamine," in accordance with the nomenclature of the period, is the α -dinaphthylguanidine of modern chemistry. But no naphthylamine was known at that time, and the possible existence of a second modification could not, in the existing state of chemical theory, have been foreseen. That the work and the Yorker found favour in the estimation of Hofmann is shown by the circumstance that on its completion he was promoted from his position of honorary assistant and made a member of the research staff, his colleague being Mr., now Professor, Arthur Herbert Church, with whom Perkin formed a friendship which lasted throughout his life. It was at this period of his career that he made that discovery of the dyestuff mauve, which for a time diverted his attention from pure to applied science, although, as is now well known, the cause of pure science was advanced at a later period by this discovery to an extraordinary degree, and in many directions quite unforeseen at the time. The story of the discovery of the first coal-tar colouring matter has been frequently placed upon record, and the fiftieth anniversary was made the occasion for an international celebration in London, in July, 1906, when Perkin became the central figure and received the homage and congratulations of chemists and technologists from every part of the world. Seldom, if ever, in the history of science has the discovery of one chemical compound of practical utility led to results of such enormous scientific and industrial importance as this accidental preparation of mauve in 1856. The details of the working out of the manufacturing process and of the methods of utilising the dyestuff belong to the history of applied science, but since the discovery was the outcome of purely scientific antecedents, and its achievement a matter which materially affected Perkin's career, it is necessary to recapitulate this chapter of his activity in the present notice.

The remarkable zeal which Hofmann's young assistant must have thrown into his work is well revealed by the circumstance

that even the activity of the Oxford Street laboratory failed to satisfy his craving for research. He was at that time kept at work on the investigations prompted by that illustrious professor whose resourcefulness appeared to be inexhaustible, and had little or no time for working independently. He accordingly fitted up, in 1854, a part of a room as a laboratory in his own home,* and there carried on his researches after the day's work at the College was over and during the vacation. It is of considerable interest to note that even at this early period his work brought him into contact with colouring matters, for, having secured the co-operation of his colleague, Mr. Church, one of the first pieces of work which they took in hand was the investigation of the products of reduction of dinitrobenzene and dinitronaphthalene. From the latter there was obtained a coloured substance which, in accordance with the prevailing views concerning the nature of such compounds, was named "nitrosonaphthyline," and a brief account of it was given to the Royal Society by Hofmann on February 5th, 1856 (*Proc. Roy. Soc.*, 8, 48), the complete description being afterwards published in the names of Perkin and Church in the *Journal of the Chemical Society (Quart. Journ.)*, 1857, 9, 6). The interest attaching to this colouring matter is that it was the first representative of the large and important group of azo-dyes derived from naphthalene ever manufactured, although its true nature was, of course, at first unknown to its discoverers, and even its ultimate composition was not accurately established at the time, because, seven years later, when Perkin and Church resumed the study of the compound, they found that it contained no oxygen, as had at first been supposed, and that it could be made more conveniently by the action of a nitrite on a salt of α -naphthylamine in the presence of alkali. The substance was re-named, in accordance with current notions, "azodinaphthyldiamine," and the amended results published by the Chemical Society (*Journ. Chem. Soc.*, 1863, 16, 207). A patent was also secured (No. 893 of 1863) † and the substance had a limited use as a dyestuff. The azodinaphthyldiamine of 1863 is the α -aminoazonaphthalene of modern chemistry, and, it may be added, is of no importance in tinctorial industry at the present time.

The discovery of a compound which happened to be a colouring matter was at this stage of Perkin's career an accidental circumstance, as was, in fact, the discovery of mauve, which was made

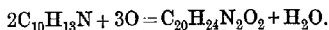
* His father's house was at that time known as "King David's Fort," Shadwell, E. The name is still preserved in King David's Lane.

† It has been pointed out by Caro (*Ber.*, 1891, 24, Appendix, p. 3) that this patent is the first claiming the production of a sulphonated azo-colour.

in this same rough home laboratory about the same time, namely, the Easter vacation of 1856. In view of the widespread notion that discoveries of industrial value are invariably the result of researches directed solely towards this practical end, it may be of interest to place once again upon record the statement that the first coal-tar colouring matter was discovered by Perkin as the outcome of as distinct a piece of pure scientific research as was possible in the light of the theoretical conceptions of that period. It must be borne in mind that in 1856 organic chemists had practically nothing to guide them in expressing the formulae of compounds but the ultimate composition derived from analytical results. It is true that the possibility of different substances having the same ultimate composition had, since the time of Wöhler and Berzelius, received recognition among chemists, but these early ideas concerning isomerism had not yet given birth to those definite conceptions of chemical structure which at a later period resulted from the application of the doctrine of valency. Thus in 1856 it was scientifically legitimate to set out from the assumption that a natural product might be synthesised if the elements composing it could be brought into combination in the right proportions. Many attempts to produce natural compounds artificially had been made on this principle since the fundamental synthesis of urea from ammonium cyanate by Wöhler in 1828, and although no success in the way of the desired syntheses can be recorded, there can be no doubt that many indirect results of lasting importance to chemical science were arrived at in this way. The discovery of mauve by Perkin is an example of such an indirect result which at first ranked as an industrial success only, and, it may now be said fortunately, for a time diverted the energies of its discoverer from the field of pure science to that of chemical industry.

In so far as the discovery of mauve is attributable to scientific as distinguished from purely technical research, it may be pointed out that in accordance with the prevailing belief that a synthetical product, if of the same empirical formula, would prove to be identical with the natural compound, Hofmann, as far back as 1849, had, as Perkin himself indicates in the Memorial Lecture (Trans., 1896, 69, 603), suggested the possibility of synthesising quinine from naphthalene, the ground for this suggestion being that the base "naphthalidine" (=naphthylamine) was at that time supposed to differ from quinine only by the elements of two "equivalents" of water, so that if the hydration of the base could by some means have been effected, quinine might be expected to be the result ("Reports of the Royal College of Chemistry," 1849, Introduction, p. 61). Ideas of this order were prevalent in the chemical world

about the middle of the nineteenth century, and Perkin has told us how, imbued with these notions, he was "ambitious enough to wish to work on this subject of the artificial formation of natural compounds" (Hofmann Memorial Lecture, *loc. cit.*). Following the method then in vogue, he came to the conclusion that the most likely generator of quinine would be allyltoluidine, since two "equivalents" of this compound, by taking up oxygen and losing hydrogen (in the form of water), would give a substance of the formula of quinine:



The experiment was tried, a salt of allyltoluidine being oxidised by potassium dichromate, but, instead of quinine, a "dirty reddish-brown precipitate" was obtained. This result, negative in one sense, still appeared of sufficient interest to the young investigator to be worth following up, and he repeated the experiment with a salt of the simpler base aniline, obtaining in this case a very dark-coloured precipitate, which, on further examination, was found to be a colouring matter possessed of dyeing properties. Thus was discovered the first of the coal-tar dyes, the subsequent and rapid development of which, from a laboratory curiosity into a technical product, brings into strong prominence the extraordinary combination of energy, skill, and resourcefulness inherent in this youth, who at the time was not much over seventeen years of age. The very fact of his continuing the investigation of what the majority of contemporary chemists would have discarded as an unpromising "Schmier," may be taken as an indication of his originality, for it must be remembered that, at that time, the main object of research in organic chemistry was to obtain definite crystalline compounds, and the formation of non-crystalline, and especially of coloured, amorphous products was considered as an indication of the failure of a reaction. This view of research method was particularly upheld in Hofmann's laboratory, and, as has frequently been pointed out by many critics of the too-rigid enforcement of this method, there can be no doubt that the discovery of the coal-tar dyes was considerably retarded by the liberal use of animal charcoal as a decolorising material. Hofmann himself, for example, is well known to have prepared rosaniline in 1858 incidentally as a by-product in the course of his study of the reaction between carbon tetrachloride and aniline, although, so far as concerned the main objects of his research, he regarded it as an impurity. To Perkin must be given the credit of having the courage to break through the traditional dislike of investigating coloured, resinous-looking products, an achievement which, in the

case of mauve, may, perhaps, be attributed to that rare combination of the scientific and artistic faculties which he was known to possess. The fact that his new product on purification gave a compound which at that time would be considered as imparting a beautiful shade of colour to fabrics when used as a dye, may fairly be claimed to have appealed to his æsthetic sense, and to have lured him on with his research, independently, at first, of immediate practical developments. Professor A. H. Church, his colleague and co-worker, has supplied the following statement with respect to this period of his career:

"It was, I think, in October, 1853, that William Henry Perkin entered the Royal College of Chemistry, and was assigned the next bench to mine in the front of the building, looking out upon the street. One year before this date I had gone through my novitiate, and had been awarded what was called a scholarship—still receiving instruction and attending the lectures, but paying no fees. Indeed, I had been carrying out from time to time some minor researches suggested by Dr. Hofmann. Perkin and I soon found we had several interests in common. We were both given to painting, and were amateur sketchers. I was introduced to his home at King David's Fort, and we began painting a picture together. This must have been soon after the Royal Academy Exhibition of 1854, when I had a picture hung. I was nearly four years Perkin's senior, but was soon impressed by his mental activity and his devotion to work.

"I remember the epoch-making experiment in which mauve was first discovered. He repeated it in my presence for my particular benefit. I distinctly recollect strongly urging him to patent his invention. Shortly after this date I left the college for Oxford, but Perkin and I were in frequent communication, and sometimes worked together after I had taken my degree in 1860, and until my appointment in 1863 to the chair of chemistry at the Royal Agricultural College.

"During the year 1855, and the spring of 1856, Perkin and I were no longer working in the same laboratory, for I had been given a bench in the professor's private laboratory on the ground floor, and was engaged in carrying out some of his most important researches of that period."

The history of the technical development of this discovery has been narrated by Perkin in his Hofmann Memorial Lecture of 1896, and it is only necessary to go through that account in order to realise the magnitude of his achievement. A youth of about eighteen, undaunted by the discouragement of his professor, the greatest living master of organic chemistry, had determined to

work out his discovery on a manufacturing scale, with no experience or training as a manufacturer himself, and with no precedent to guide him in the construction of plant for carrying on operations, which had, up to that time, never been conducted on more than a laboratory scale. Hofmann's opposition to his young assistant's leaving the paths of pure science, and embarking upon what, no doubt, appeared to his maturer judgment a most risky undertaking, is quite understandable, and fully justifiable. Everything in connexion with the new industry had to be worked out from the very beginning—the methods for the isolation and preparation of the raw materials, as well as the manufacture of the new dyestuff, and the prejudices of the dyers and printers against innovation had also to be overcome. With all this responsibility ahead of him, Perkin, encouraged, no doubt, by the favourable report concerning the dyeing qualities of his new product furnished by certain practical dyers, and especially by Messrs. Pullar, of Perth, formally resigned his position at the Royal College of Chemistry, and boldly entered upon his career as an industrial chemist. He has touchingly placed upon record his indebtedness to his father, who, although, as already stated, at first inclined to be adverse to his taking to chemistry as an occupation, had, at the time of the discovery of mauve, so much confidence in his son's ability that he threw in his lot with the new venture, and devoted the greater part of his life's savings to the building of a factory, for which a site had been secured at Greenford Green, near Sudbury, at which latter place Perkin afterwards resided. His elder brother,* Thomas D. Perkin, *viz.*, during the summer vacation of 1856, had assisted in making mauve in the laboratory on a somewhat larger scale, in order to supply specimens for testing by the dyers, also joined in the undertaking. A patent was secured (No. 1984, August 26th, 1856), and the building of the works commenced in June, 1857, and six months later the new dyestuff, under the name of "Aniline Purple," or "Tyrian Purple," was being manufactured in sufficient quantity to supply one of the London silk dyers.† The subsequent development of this precursor of the coal-tar dyes forms an interesting and, indeed, a romantic chapter in the history of applied science. Its reputation spread rapidly; from silk dyeing its application was extended to cotton dyeing and to calico printing, and at every stage of a career which may be fairly described as triumphant, the master hand of William Henry Perkin can be detected. Now we find him working out processes for the manu-

* Born 1831, died 1891.

† The name "Mauve," by which it was afterwards generally known, was given to the dyestuff in France.

facture of nitrobenzene and aniline on a scale never before attempted, then we learn of his introducing improvements into the methods of silk dyeing on the large scale, and of his discovering suitable mordants for enabling the dyestuff to be applied to cotton fibre both by dyers and calico printers. Well may it be said in Perkin's own words: "In fact, it was all pioneering work."*

In spite of these splendid pioneering efforts, however, it seems that the recognition of the value of the product at first took place but slowly in this country, and it was not until it had been taken up in France that its merits for tinctorial purposes became generally recognised. In a private communication addressed to the writer of this notice on April 3rd, 1906, Perkin states: "The value of the mauve was first realised in France, in 1859. English and Scotch calico printers did not show any interest in it until it appeared in French patterns, although some of them had printed cloth for me with that colour." The "Société Industrielle de Mulhouse," it may be added, awarded him a silver medal for his discovery in 1859, and afterwards a gold medal.† It is of interest to note also that a paper was read by him at the Leeds meeting of the British Association in 1858, under the title, "On the Purple Dye obtained from Coal Tar" (Reports, 1858, p. 58), when specimens of the substance and fabrics coloured by it were exhibited. No more appropriate place than this town, in the centre of one of the chief seats of the tinctorial industry in Great Britain, could possibly have been selected for bringing the discovery under the notice of chemists and technologists. Sir John Herschel was President of the Chemical Section, and, by a remarkable coincidence, in the opening address of the President of the Association, Professor (afterwards Sir Richard) Owen, there occur the following passages *à propos* of the general progress of organic chemical synthesis: "To the power which mankind may ultimately exercise through the light of synthesis, who may presume to set limits? . . . Already, natural processes can be more economically replaced by artificial ones in the formation of a few organic compounds. . . . It is impossible to foresee the extent to which chemistry may ultimately, in the production of things needful, supersede the present vital agencies of nature." This pronouncement at the meeting when the first of the coal-tar colouring matters was exhibited—

* Speech at the Jubilee Banquet in New York, October 6, 1906. See also the Hofmann Memorial Lecture, *loc. cit.*, p. 603.

† The impetus given to the new colouring matter through French influence was also referred to by Perkin in his reply to Professor Haller at the Jubilee Meeting in 1906 (Report, p. 11); see also *Journ. Society of Dyers and Colourists*, April, 1907, p. 106.

discovery which laid the foundations of an industry which now supplies as tar products the colouring matters of madder and indigo—may be looked upon as prophetic.

The influence of this inaugural work by Perkin upon the subsequent history of the industry is too well known to need recapitulation. It is only necessary to point out that the introduction of aniline—at that time a mixture of homologues—into the market soon led other investigators to enter the field of colour chemistry, and new dyestuffs made their appearance in rapid succession, the most noteworthy after mauve being magenta, which was discovered as a technical product in 1859 by Verguin, and manufactured for a short period by his process* by the firm of Renard Frères et Franc, of Lyons. In fact, the stream of competition in the course of a few years turned against the original mauve, the demand for which gradually fell off as other colouring matters of a similar or brighter hue were introduced. The consideration of chief interest in connexion with Perkin's successful venture into the domain of applied chemistry is, however, from the present point of view, the influence which his work in this field exerted upon pure science. That it has exerted an enormous influence is now generally recognised, and a critical examination of the course of development of the industry will show that the gain by chemical science has been of a twofold character—a direct and an indirect gain.

In the first place, as the direct result of introducing into commerce in large quantities organic chemical products which had before been but laboratory curiosities, a great stimulus was given to research, and chemical workers of the highest repute took up the investigation of the new products, both raw materials and colouring matters. As an indirect consequence, also, many new compounds of industrial value were discovered incidentally in the course of manufacturing operations conducted on the large scale, and these, with the colouring matters which from time to time appeared as novelties, furnished endless subject matter for research, the results so obtained often proving of the greatest scientific importance. Not the least interesting circumstance in connexion with this chapter of chemical history is the fact that Hofmann himself soon entered the field of tinctorial chemistry, to which he made many contributions of the utmost value both from the scientific and technological point of view. He was, in fact, for many years recognised as the leading scientific authority on coal-tar colouring matters, and many of his discoveries were practically

* By heating crude aniline (*i.e.*, aniline containing toluidine) with stannic chloride.

utilised in the factories. Then, again, there can be no doubt that the success of the new industry and the succession of important scientific discoveries which followed its development attracted large numbers of students into the chemical schools, and many gifted and active workers were by this means drawn as recruits into the ranks of scientific chemists. It is, indeed, not going too far to say that the discovery of the coal-tar colouring matters brought about such a revival in the study of organic chemistry, and particularly in that of the so-called "aromatic" series, that when the epoch-making conception concerning the constitution of these compounds had been given to the world by Kekulé in 1865, the rapid extension of the "benzene theory" was enormously facilitated by the resources which the new industry had given to pure science. If it is true that the new theory materially advanced the cause of the industry, it is no less true that the industry contributed to the advancement of the theory, the verification of which might have been delayed for a generation or more without such support. No better illustration of the interdependence of science and industry has ever been given to the world than this particular example of the action and reaction between theoretical and applied chemistry.*

The success of the new industry not only reacted upon the science of chemistry in the way indicated, but it may be claimed that, contrary to Hofmann's forebodings, it proved in the long run beneficial in every way to Perkin himself, and through him to that science to which he devoted his life. He has told us that when, being fully convinced of the value of mauve, he announced his intention of leaving the College of Chemistry and taking up the manufacture of the new colouring matter, he determined not to allow the manufacturing career to check his research work, and nobly did he adhere to his resolution. His published papers show that in spite of all his technical work the stream of original investigation was never allowed to stagnate. Only a year after the starting of the Greenford works, namely, in 1858, in conjunction with Duppa, he discovered that aminoacetic acid or "glycocoll," a compound which up to that time had only been prepared by the

* The consideration of the later important influence upon other branches of science arising, often in most indirect and unforeseen ways, from the applications of coal-tar products to such subjects as bacteriology, histology, therapeutics, photography, etc., would swell this notice to an inordinate extent. Although results of incalculable value have been achieved in these fields, Perkin himself is not particularly identified with any of the lateral developments of his initial pioneering labours. References to this aspect of the subject were made in some detail at the Jubilee celebration in 1906. (See the official Report published by the Memorial Committee, and also a paper by Dr. Hugo Schweitzer in *Science*, No. 616, October 19, 1906, p. 481.)

decomposition of natural products, could be obtained by heating bromoacetic acid with ammonia.* A general survey of his work during his connexion with the coal-tar colour industry, which ceased in 1874, brings out very clearly the double line of thought which during that period actuated his research work. Concurrently with the investigation of the dyestuffs, he carried on researches in other departments of organic chemistry which had at that time no relations with tinctorial chemistry. Thus we find that by 1860 he, in conjunction with Duppa, had discovered the relationship between tartaric and fumaric-maleic acid, and had effected the synthesis of racemic acid from dibromosuccinic acid, a line of work which was followed up with signal success (Perkin and Duppa, *Annalen*, 1860, 115, 105; *Quart. Journ. Chem. Soc.*, 1860, 13, 102; Perkin, *Journ. Chem. Soc.*, 1863, 16, 198; Perkin and Duppa, *Annalen*, 1864, 129, 373; Perkin, *Proc.*, 1888, 4, 75). About 1867 he must have commenced those researches on the action of acetic anhydride upon aromatic aldehydes which led to such important developments, and culminated in that beautiful method of synthesising unsaturated acids now known as the "Perkin synthesis." The first paper of this series bore the title, "On the Action of Acetic Anhydride upon the Hydrides of Salicyl, Ethylsalicyl, &c." (*Journ. Chem. Soc.*, 1867, 20, 586), and as the outcome of this work the synthesis of coumarin, the odorous substance contained in Tonka Bean, etc., was announced the following year ("On the Artificial Production of Coumarin and Formation of its Homologues," *Journ. Chem. Soc.*, 1868, 21, 53 and 181). The production of a vegetable perfume from a coal-tar product was thus first made possible by Perkin, and the continuation of this work, after his retirement from the industry, led to his celebrated discovery of the synthesis of cinnamic acid from benzaldehyde, an achievement which subsequently, in the hands of Adolf v. Baeyer and H. Caro, made possible the first synthesis of indigo from tar products.† It is of interest to note also that while still in the coal-tar colour industry he took part in the discovery of synthetical methods for producing glyoxylic acid from dibromoacetic and

* Perkin and Duppa, *Annalen*, 103, 112. This discovery is specially referred to, not only as illustrating Perkin's extraordinary activity during this busy period, but also because the compound is the type of a large group of amino-acids which of late years have become of extreme importance owing to their relationship to the proteins, as shown by Emil Fischer and his co-workers.

† "A Preliminary Notice of the Formation of Coumarin, Cinnamic Acid, and other similar Acids," *Chem. News*, 1875, 32, 258; "On the Formation of Coumarin and of Cinnamic and of other Analogous Acids from the Aromatic Aldehydes," *Journ. Chem. Soc.*, 1877, i, 388.

bromoglycollic acids, thus giving the first insight into the constitution of glyoxylic acid, a result of considerable significance in view of the important part attributed by many modern chemists to this acid in the photosynthetic processes going on in growing plants (Perkin and Duppa, *Journ. Chem. Soc.*, 1868, 21, 197).

The research work done during Perkin's colour-making period was carried on in a laboratory in a house just outside the Greenford factory, where also the scientific investigations in connexion with the colouring matters were conducted, the double line of work already indicated being revealed by the papers published during that period. It has not been considered necessary to give a complete list of these papers in the present notice, but it will be of interest to call attention to the fact that the purely scientific study of the colouring matters undertaken at this time centred round his early discoveries. It was in this new laboratory at Greenford that he and Church continued the investigation of "azodimaphthylidiamine" already mentioned, and discovered a method for resolving this compound by complete reduction, thus introducing a process which is still the standard one for determining the constitution of azo-compounds, and at the same time leading to the isolation of the first diamine derived from naphthylamine (*Journ. Chem. Soc.*, 1865, 18, 173). Nor did he allow his scientific interest in his first discovered dyestuff to flag, for one paper on mauve from the purely chemical point of view was published during his connexion with the industry and another after his retirement in 1874.*

In 1868 it was shown by Graebe and Liebermann that the colouring matter of the madder, alizarin, one of the most ancient of vegetable dyestuffs and a substance of immense value for tinctorial purposes, was a derivative of the coal-tar hydrocarbon anthracene, and not, as had up to that time been believed, a derivative of naphthalene. The synthesis of this compound was effected by Graebe and Liebermann in that year, and patents for its manufacture from anthracene secured in Germany and in Great Britain, this being the first instance of a natural vegetable colouring matter having been produced artificially by a purely chemical method. This discovery had a great influence upon Perkin's career as an industrial chemist, and may, indeed, be considered to have marked a new phase of his activity in this field. There was no

* "On Mauve or Aniline Purple," *Proc. Roy. Soc.*, 1868, 12, 713 (abstract); 864, 12, 170 (full paper). "On Mauveine and Allied Colouring Matters," *Trans.*, 879, 35, 717. In 1861 he lectured before the Chemical Society on the new coal-tar colouring matters, on which occasion, he has told us, Faraday was among his auditors and congratulated him at the end of the lecture.

living worker in this country at that time besides Perkin who so completely combined in himself all the necessary qualifications for taking advantage of such a discovery. Imbued with the spirit of his early ambition to produce natural compounds synthetically, with more than a decade's experience as a manufacturer, with the resources of a factory at his disposal, and, not least, with special experience of anthracene as the very substance upon which, at Hofmann's instigation, he commenced his career in research work, it can readily be understood that Graebe and Liebermann's results should have appealed to him with special significance. The first patented process of the German discoverers was confessedly too costly to hold out much hope of successful competition with the madder plant, requiring as it did the use of bromine. Perkin at once realised the importance of cheapening the process by dispensing with the use of bromine, and undertook researches with this object. As a result, the following year (1869) witnessed the introduction of two new methods for the manufacture of artificial alizarin. In one of these processes dichloroanthracene was the starting point, and in the other the sulphonic acid of anthraquinone, the first being of special value in this country owing to the difficulty of obtaining at that time "fuming" sulphuric acid in large quantities. The second process, which is the one still in use, had quite independently been worked out in Germany by Caro, Graebe, and Liebermann, and patented in England practically simultaneously with Perkin's.* The subsequent industrial development of this brilliant achievement has now become historical; the artificial alizarin has completely displaced the natural colouring matter, and madder growing as an industry has become extinct. It is of interest, as showing the growth of the new industry, to reproduce Perkin's statement in 1876:

"The quantity of madder grown in all the madder-growing countries of the world, prior to 1868, was estimated to be 70,000 tons per annum, and at the present time the artificial colour is manufactured to an extent equivalent to 50,000 tons, or more than two-thirds of the quantity grown when its cultivation had reached its highest point" (Presidential Address to Section B of the British Association, Glasgow, 1876, "Reports," p. 61).

The development of this branch of the coal-tar industry in the Greenford Green Factory has also been recorded by Perkin:

"Before the end of the year (1869) we had produced 1 ton of this colouring matter in the form of paste; in 1870, 40 tons; and in 1871, 220 tons, and so on in increasing quantities year by

* The patents are, Caro, Graebe, and Liebermann, No. 1936, of June 25, 1869, and W. H. Perkin, No. 1948, of June 26, 1869.

year . . . up to the end of 1870 the Greenford Green Works were the only ones producing artificial alizarin. German manufacturers then began to make it, first in small and then in increasing quantities, but until the end of 1873 there was scarcely any competition with our colouring matter in this country" (Hofmann Memorial Lecture, Trans., 1896, 69, 632).

This brilliant achievement in technology again served to bring out the purely scientific spirit which animated all Perkin's work. The chemical investigation of anthracene derivatives was carried on concurrently with the industrial development of the factory process, and also after his retirement, about a dozen papers on these compounds having been published between 1869 and 1880. The discovery of a practical process for the manufacture of alizarin thus led to the utilisation of another coal-tar hydrocarbon anthracene, which had up to that time been a waste product, and the methods for isolating and purifying this substance had, as in the case of benzene, etc., to be worked out in the factory. All the difficulties inseparable from large-scale operations with new materials were successfully surmounted by Perkin; the increasing demand for artificial alizarin taxed all the resources of the factory, and by 1873, when the necessity for introducing enlarged plant became imperative, advantage was taken of the opportunity for transferring the works to the firm of Brooke, Simpson, and Spiller, the successors to the firm of Simpson, Maule, and Nicholson, which had co-operated with Perkin in the early days of the mauve manufacture. The later history of the works is referred to in the technical portion of this notice.

On completion of the sale of the Greenford Green Works in 1874, Perkin retired after eighteen years' connexion with the industry. In view of the enormous development of this branch of manufacture in later times, it is of interest to recall the circumstance already mentioned that the whole output of the original factory, both in number and quantity of products, would appear quite trivial in comparison with that of one of the great German factories now in existence—a fact which only serves to emphasise the extraordinary fertility of the seed originally planted by Perkin, whose labours as a technologist led, as a practical issue, to the acquisition of sufficient means to enable him to withdraw altogether from the industrial side of chemistry at the comparatively early age of 36, while still in the prime of life. By many who have watched the decadence of the coal-tar colour industry in this country, he has been blamed for cutting himself so soon adrift from his own offspring. There is no doubt that the life of the industry here would have been prolonged if he had kept in touch with it, but it must not

be forgotten that at the time of his retirement he left things in a very flourishing condition. Other factories had developed into successful establishments, and Great Britain was well to the front in this branch of manufacture. Neither Perkin nor his contemporaries could have foreseen in 1874 that our position would later be so successfully assailed by foreign competitors. To a man with his most moderate personal requirements, and with the ardour of the original investigator unquenched, the means of retirement—modest enough as compared with the fortunes accumulated by modern successful manufacturers—simply meant the opportunity of giving practical effect to that resolution concerning his mission as a research chemist which he had formed as a youth, which he had adhered to throughout his industrial career, and which it was his desire to carry out untrammelled by business distractions throughout the remainder of his working period.* Industry may, and no doubt did, lose by his decision, but science gained by thirty years of his activity from the period of his retirement down, practically, to the end of his life.

The contributions to chemical science which proceeded from Perkin's laboratory after 1874 have, to some extent, been referred to. After his connexion with the Greenford Green Factory had terminated, he had a new house built at Sudbury, converting the adjacent house in which he had previously resided into a laboratory, and it was here that from 1875 he continued his investigations of those colouring matters with which his manufacturing experience had brought him into contact, such as mauveine, the anthracene derivatives, etc. In 1881 he first drew attention to a certain physical property of some of the compounds which he had prepared, namely, their magnetic rotatory power, which observation diverted his activity into an entirely new channel. On further development in his hands this method became a powerful weapon in dealing with questions of chemical constitutions, and the remainder of his life was more or less devoted to its elaboration. As Perkin's name must always be intimately associated with this chapter of physical chemistry, it will be of interest to place upon record his earliest observation. In a paper entitled "On the Isomeric Acids obtained from Coumarin and the Ethers of Hydride of Salicyl" (*Trans.*, 1881, 39, 409), he describes the methyl ether of " α -methylorthoxy-phenylacrylic acid," which he had first prepared in 1877, and in this paper occurs the statement:

* "The great importance of original research has been one of the things I have been advocating from the commencement of my chemical career, in season and out of season."—From a speech by Perkin at the Jubilee Banquet in London, on July 26, 1906.

A determination of its magnetic rotary power gave for the yellow ray 2.334, water being taken as 1. Test observations were made at the same time with water and carbon bisulphide, and gave results very nearly identical with those obtained by Becquerel" (*Ann. Chim. Phys.*, 1877, [v], 12, 22; *loc. cit.*, p. 411).

It is not difficult to follow, at least conjecturally, the mental process by which Perkin was enabled to foresee that this property might be utilised for investigating the constitution or structure of chemical molecules, a subject which even at that time was beginning to bristle with difficulties and ambiguous results when handled by purely chemical methods. He had for precedent the success which had attended the study of other optical properties of organic compounds, such as ordinary (not induced) rotatory power, dispersion, refractivity, etc., and he threw himself seriously into this line of work, armed with the skill of an accomplished experimenter, and with that true instinct as a chemist which enabled him to deal with his materials in such a manner that his results at once commanded complete confidence, in spite of the circumstance that this kind of work was for him a totally new departure. In 1882 he published a preliminary paper on the application of this method, and a complete account in 1884.*

From that time onwards the Chemical Society received and published constant instalments of his work, the fertility of the method being shown, not only by the long list of papers published in his own name, but also by the numerous observations recorded in the papers of other workers, to whose service his apparatus and his observational powers were frequently and ungrudgingly devoted. His achievements in this field are well summarised in a letter from Professor J. W. Brühl, of Heidelberg, himself one of the pioneers in the application of optical methods for the determination of chemical constitution, sent to the writer of this notice for transmission to Perkin on the occasion of the Jubilee celebration in 1906: "Availing yourself of the marvellous discovery of your great countryman, Michael Faraday, you undertook to investigate the relations between the chemical composition of bodies and their magnetic circular polarisation—that is to say, one of the general properties of all matter. Before you began work there was little, almost nothing, known of this subject, certainly nothing of practical

* "On Rotatory Polarisation by Chemical Substances under Magnetic Influence," *Trans.*, 1882, 41, 330. "On the Magnetic Rotary Polarisation of Compounds in Relation to their Chemical Constitution; with Observations on the Preparation and Relative Densities of the Bodies examined," *ibid.*, 1884, 45, 421. This last paper, which occupies 60 pages of the volume, contains a full description of the apparatus and method of observation.

use to the chemist. You created a new branch of science, taught us how, from the magnetic rotation, conclusions can be drawn as to the chemical structure of bodies, and showed that the magnetic rotation allows us to draw comprehensive and certain conclusions as to the chemical constitution of substances, just as we may from another general physical property, viz., refraction and dispersion. And by showing that both these physical methods of investigation lead to completely harmonious results, you did essential service to both the branches of study, and also to chemistry, which they are destined to serve."

This last statement by Brühl, which relates to one of the most interesting results of the study of magnetic rotation, has reference to a development of Perkin's work which brought him into association with the late John Hall Gladstone, the pioneer and leading authority in this country at that time on the relations between refractive and dispersive power and chemical constitution. The correspondence between the results arrived at by these two optical methods forms the subject of a joint paper by Gladstone and Perkin published in 1889.* Eighteen years later Perkin's last paper, to which attaches the melancholy interest that it was read before the Chemical Society on April 18th, 1907, only a few months before his death, bears the title: "The Magnetic Rotation of Hexatriene, $\text{CH}_2\text{:CH}\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}_2$, and its Relationship to Benzene and other Aromatic Compounds: also its Refractive Power" (Trans., 1907, 91, 806).

Although, as already stated, the latter part of Perkin's life was devoted mainly to his work on magnetic rotation, he published also during this period a few papers relating to other subjects, among which perhaps the most notable is his contribution to the subject of low temperature combustion, entitled "Some Observations on the Luminous Incomplete Combustion of Ether and other Organic Bodies" (Trans., 1882, 41, 363). The writer of this notice well remembers the keen interest with which the experiments were followed in the darkened meeting-room of the Chemical Society at Burlington House when this paper was read. In view of the

* "On the Correspondence between the Magnetic Rotation and the Refraction and Dispersion of Light by Compounds containing Nitrogen," Trans., 1889, 55, 750. The correspondence between Perkin and Gladstone during this period has been placed at the disposal of the writer by Miss Gladstone. The letters are interesting as showing the extreme conscientiousness in every detail with which Perkin carried out his work. The results are embodied in the above paper, and a further contribution by Perkin was published two years later, under the title, "The Refractive Power of certain Organic Compounds at different Temperatures," Proc., 1891, 7, 115. In his later papers he dealt with refractivity as well as magnetic rotation (Trans., 1890, 69, 1; *ibid.*, 1900, 77, 267, etc.).

modern revival in the scientific study of the chemical mechanism of combustion, it is of importance that Perkin's observations should not be allowed to fall into oblivion.

It has been claimed in a previous part of this notice that Perkin's entry into the domain of chemical industry was no real loss, but actually a gain to pure science. His published papers, considered in detail, show that his contributions to "colour chemistry" are far outweighed by his work in other fields. In fact, the extension and completion of the investigation of the dyestuffs of his industrial period is due to other workers, and Perkin's achievements in this direction are, on the whole, more of a technological than of an abstract scientific character, the constitution of most of the colouring matters having been subsequently worked out chiefly by the group of brilliant Continental investigators attracted by the success of the new industry, and stimulated by the rapid development in chemical theory then going on in Germany.* But although Perkin was overshadowed by his own achievements as a "colour chemist" by his subsequent career, the whole success of his life, and the inestimable gain which chemical science has derived from his labours, must be directly attributed to his industrial undertakings, for it may safely be asserted that had he not been rendered independent by the success of the Greenford Green Factory, he would never have found an opportunity for that continuous devotion to research which is so essential for the achievement of results of lasting value. Having determined in early life to adopt chemistry as a career, he would of necessity have been compelled to become either a manufacturer or to have entered an educational establishment. In the former capacity he would, no doubt, have succeeded, but in any subordinate post he might have spent long years before acquiring independence. As a teacher his prospects of making a position at the time of his connexion with the Royal College of Chemistry were most slender. There were but very few posts which he could have filled; originality as an investigator was of minor importance as a qualification for the teaching profession, and the stamp of university training was generally considered absolutely essential for holding any important appointment in that profession. Perkin's any minor teaching post would have been lost to science. Happily the comparatively rapid financial success of his early discoveries placed him in that category which comprises such names as Cavendish, Herschel, Joule, Murchison, Spottiswoode, Lyell, and

*For example, the constitution of mauveine was established broadly by Fischer and Hepp about 1890; that of the colouring matters of the rosaniline group (magenta, methyl-violet, etc.), by E. and O. Fischer, about 1878, and that of uranine about 1883 by Nietzki.

Darwin—representatives of that band of independent devotees of science who have more than any other class helped to maintain the prestige of this country. Truly may it be said that to a man of *his temperament success as a manufacturer meant salvation as an original worker.*

Reviewing Perkin's scientific work as a whole, its chief characteristic is its solidity. His mind was not of that order which readily entered into the region of speculation; he was a typical representative of that school of chemists to whom the conscientious accuracy of experimental facts is of primary importance—the school which has laid those solid foundations of chemical science upon which all superstructures of theory must be erected. It is for this reason that it may be predicted with certainty that his work will live in the history of modern chemistry whatever changes in theoretical conceptions the future may have in store. He himself witnessed with the progress of the science radical changes in the views of chemists concerning the mechanism of the reactions or the nature of the compounds which he had discovered. With true philosophic spirit he accepted the evidence of other workers and welcomed the legitimate development of his own discoveries. Whatever modification of theory may have been rendered necessary by the accumulated labours of the great and ever-growing army of investigators which he lived to see following the tracks which he had been the first to tread, it may be safely asserted that his own early footprints have been, and always will be, ineffaceable.

Perkin was by disposition a man of extreme modesty and of a most retiring nature. His devotion to science and the domesticity of his character accounted so completely for his time that, beyond participating in the administrative work of the scientific societies with which he was connected, he took but little part in extraneous affairs. He was not particularly of a business turn of mind in the commercial sense, and during his industrial career his brother Thomas was the chief man of business connected with the factory. One line of work distinct from his purely scientific occupations is, however, worthy of special record, because it enabled him to exert some influence in the cause of technical and scientific education. His family had for a long period been connected with the Leather-sellers' Company, and through this connexion he was enabled to promote the cause of chemical research and also to become, as the representative of his Company, a member of the governing body of the City and Guilds of London Institute, whose meetings he attended with considerable regularity, although, unless specially appealed to, he seldom took part in the discussions at the Council

able. But his influence in the City of London, although unobtrusive, was of a most beneficial character, and every movement for the promotion of science and of scientific education was certain to receive his support. His special knowledge of the requirements of the chemical technologist and his sympathy with the teaching staffs have contributed in no small degree to promote the cause of sound chemical education in London through the City and Guilds Institute. As an illustration of the modesty of his character, it may be of interest to relate that many of his colleagues in the City were unaware, until the Jubilee of 1906, that the William Perkin who sat at their meetings was the same man who, half a century before, had laid the foundations of a great industry. The following details concerning his connexion with the leathersellers have been supplied by the late Mr. W. Arnold Leppburn, the Clerk to the Company:

"William Henry Perkin, son of George Fowler Perkin, was made free by patrimony, November 13th, 1861.

"George Fowler Perkin, son of Thomas Perkin, was made free by patrimony, February 4th, 1829.

"Thomas Perkin, apprenticed to Isaac Roberts, March 16th, 1772, was made free by servitude, July 7th, 1790.

"William Henry Perkin served the office of Steward, 1881-2; 4th Warden, 1885-6; second Warden, 1895-6; Master, 1896-7.

"During the Mastership of Dr. Perkin in 1896 the Company, at his instance, resolved to found a Research Fellowship in Chemistry as applied to Manufactures, tenable at the Central Technical College of the City and Guilds Institute, and to grant £150 a year in support thereof."

A portrait of Perkin in his robe as LL.D. of the University of St. Andrews, painted by Henry Grant in 1898, is on the wall of the Leathersellers' Hall in St. Helen's Place.

Although his single-minded devotion to his researches and his retiring nature caused Perkin to remain in comparative obscurity from the point of view of the general public, his real worth was well known to, and received frequent recognition from, his scientific colleagues. In this respect his history is that of the majority of active workers in the field of science in this country who do not wield the pen as *littérateurs*, or whose achievements are not of a sufficiently startling kind to create public notoriety. With the passing of the generation which witnessed the interest aroused by the discovery of mauve, and which was fanned into temporary excitement by the sensational accounts circulated by the news-

papers of the period, the memory of Perkin faded from the public mind. To most of his fellow countrymen the memorable international gathering in London in 1906 came as a revelation that they could claim as their compatriot the man whom all the nations had sent their representatives to honour as an individual, and in celebration of the fiftieth anniversary of the discovery of the first of the synthetic dyestuffs.

Perkin was elected into the Royal Society in 1866; he served on the Council in 1879-81, and again in 1892-94. In 1893-94 he was made one of the Vice-Presidents. He joined the Chemical Society in 1856, served on the Council in 1861-62, and in 1868-69; was Secretary from 1869 to 1883, and President from 1883 to 1885. By way of academic distinctions he received the degree of Ph.D. from the University of Würzburg in 1882; the degree of LL.D. from the University of St. Andrews in 1891; and was made a D.Sc. of Victoria University in 1904. In connexion with the Jubilee of 1906, the University of Heidelberg conferred upon him the degree of Ph.D., the Munich Technical High School awarded him the diploma of Dr. Ing., and the same year the Universities of Oxford and Leeds gave him the degree of D.Sc. During his subsequent visit to America in the autumn of 1906, in connexion with the celebrations organised in that country, he received the degree of D.Sc. from Columbia University, and LL.D. from the Johns Hopkins University, of Baltimore, the latter degree having been most appropriately conferred by his chemical colleague, President Ira Remsen.

He was President of the Society of Chemical Industry in 1884-85, at the time of his death was President of the Society of Dyers and Colourists,* and had recently accepted office as President of the Faraday Society. In 1884 he was made an Honorary Foreign Member of the German Chemical Society. Following the early recognition of his technological work by the "Société Industrielle de Mulhouse," already referred to, he received from the Royal Society a Royal Medal in 1879, and the Davy Medal in 1889; from the Chemical Society the Longstaff Medal in 1888; from the Society of Arts the Albert Medal in 1890; from the Institution of Gas Engineers the Birmingham Medal in 1892, and the Gold

* In honour of the founder of the industry this Society has established a Perkin Medal "for inventions of striking scientific or industrial merit, applicable to, or connected with, the tinctorial industries." Perkin's last official act in connexion with this Society was to accompany a deputation to the Dyers' Company asking the latter to contribute towards the foundation of a prize for the encouragement of research in tinctorial chemistry. The American Memorial Committee also founded a Perkin medal for American chemists in 1906 in connexion with their Jubilee Celebration in New York.

Medal of the Society of Chemical Industry in 1898. At the Jubilee Celebration in 1906, Professor Emil Fischer, on behalf of the German Chemical Society, presented him with the Hofmann Medal, and Professor Haller, on behalf of the Chemical Society of Paris, with the Lavoisier Medal.

The influence which Perkin has exerted upon this generation is not to be measured solely by his achievements in pure and applied chemistry. His life was noble in its simplicity, and his single-minded devotion to his work, combined with a character known to be religious in the highest and best sense of the term, will bequeath to posterity an enduring example of humility in the face of success which would have marred many men of smaller moral calibre. The financial success of his early manufacturing experience was turned to account simply as a means of advancing science, and no distinction which he ever gained throughout a career which culminated in 1906, when the King conferred upon him the honour of Knighthood, and when the nations of the world assembled to render him homage, had the slightest influence upon the modesty and gentleness of his disposition. It was his personality that caused him to be revered in his domestic circle, and to be beloved by all who enjoyed the privilege of his friendship. Two of the addresses presented at the Jubilee meeting in 1906 give striking expression to the universal esteem in which he was held as a man:

"But however highly your technical achievements be rated, those who have been intimately associated with you must feel that the example which you have set by your rectitude, as well as by your modesty and sincerity of purpose, is of chiefest value." (From the address presented by the Chemical Society.)

"You have given to science the allegiance of a noble life, and you have not allowed the seductions of wealth to abate the loyalty of your devotion to truth and knowledge. This is an example for which the age owes you unstinted thanks. . . . Amid these varied activities it is pleasant to know that you have cultivated the full humanity of life. Music and art have found in you a devoted disciple, and in the family and social relationship of life you have shown that science gives a truer interpretation of, and a deeper meaning to, all that is sacred and good in the heart of man." (From the address presented by the Society of Dyers and Colourists.)

Perkin was twice married, his first wife being a daughter of the late Mr. John Lisset; some years after her death he married the daughter of Mr. Herman Mollwo. Lady Perkin, three sons, all of whom have made their mark as chemists, and four daughters survive. Two of his sons, William Henry and Arthur George, were elected into the Royal Society in 1890 and 1906 respectively, and

it was always a source of great satisfaction to him to know that all his sons were following in his footsteps. In his general mode of life Perkin was a man of extreme frugality, robust and active to the last. To one of his retiring habits the strain accompanying the Jubilee celebrations in 1906 and the subsequent ordeal of his American tour must have been considerable, but he bore all the excitement and fatigue without the least indication of discomfort. Literally he died in harness; a few months previously he had read his last paper before the Chemical Society, and he was looking forward to being able to resume his research work quietly and uninterruptedly after the distractions of 1906. The illness which brought his noble and useful life to an end, which, in view of his activity, cannot but be regarded as premature, did not at first reveal any serious symptoms. The writer of this notice was with him a few hours before his death, and although he complained of suffering pain he spoke hopefully of his condition and anticipated being soon able to leave his room. The illness proved, however, to be more serious than he or his family were aware of; a sudden change for the worse occurred, and on July 14th, 1907, he passed away in perfect peace and in the full tide of well-won honour.

TECHNICAL ASPECTS OF PERKIN'S DISCOVERY OF MAUVE.

In dealing with the technical development of Perkin's discovery it is of interest to consider in the first place the state of affairs with respect to the raw materials required for the manufacture of mauve. These were benzene, nitrobenzene, and aniline.

Benzene was discovered by Michael Faraday, in 1825, as a component of the liquid obtained by the compression of oil-gas. Twenty years later Hofmann found this hydrocarbon in coal-tar, and proved its presence by preparing from it nitrobenzene and aniline, the latter being identified by the usual tests. The occurrence of benzene in coal-tar was thus known in 1845, and in 1848 one of Hofmann's brilliant young students at the Royal College of Chemistry, Charles Blachford Mansfield, at the instigation of his illustrious master, undertook a systematic study of coal-tar, with a view to the isolation and identification more especially of the "neutral liquid oils," of which he tells us in his paper published by the Chemical Society in 1849 we had at that time "no precise information." When Mansfield took up this work, a few definite compounds were known to exist in this tar, notably naphthalene, which had been isolated by Garden in 1820, and certain acid and basic substances, such as phenol (carbolic acid), aniline (kyanol), quinoline (leucol or leucoline), and pyrrole, all of which had been

isolated by Runge in 1834. Anthracene, under the name of "paraphthaline," was isolated by Dumas and Laurent in 1833, although it is now known that their original analysis, which assigned to this hydrocarbon 15 atoms of carbon, was erroneous. Chrysene and pyrene had also been indicated, but only superficially studied by Laurent in 1837. To the basic constituents, picoline was added in 1846 by Anderson.

Such was the state of knowledge when Hofmann set Mansfield to work upon the coal-tar hydrocarbons. The paper embodying his results is entitled, "Researches on Coal Tar. Part I.,"* and now, nearly sixty years after its publication, it can still be read with interest and profit. Its contents have become historic in connexion with the colour industry, and must rank with Runge's celebrated papers of 1834 (*Pogg. Annalen*, **31**, 65, 513; **32**, 308, 328) among the most important contributions to tar chemistry that preceded the foundation of that industry. The still devised by Mansfield or fractionally distilling the tar oils embodied the "reflux" principle of our modern rectifying columns. In the way of definite products he isolated and characterised benzene with considerable precision; he found that it could be purified by fractional distillation and by crystallisation at a low temperature. It is of interest to note in passing that the analysis of the hydrocarbon was made for him by Edward Chambers Nicholson, another of Hofmann's pupils, who at a later period played a very conspicuous part in connexion with the coal-tar colour industry of this country. Of the higher boiling-point hydrocarbons, he also isolated toluene and two of the higher homologues, which he was inclined to identify with cumene and cymene respectively. It is now known that the fraction which he considered to be cumene was xylene, and it is very doubtful whether cymene is contained in coal-tar at all. There can be no doubt that he had not individual compounds to deal with in the case of these higher homologues, and it was evidently his intention to have continued the investigation in this direction, as the paper is entitled "Part I."

Unfortunately, the author never lived to complete his work. A few years after the publication of this first paper, he met with an accident through the ignition of some hydrocarbons which he was distilling, and was burnt so severely that he died in the thirty-fifth year of his age. The late Mr. Robert Holliday informed the writer some years ago that Mansfield was at that time carrying on experiments in London with coal-tar hydrocarbons for their

* *Quart. Journ. Chem. Soc.*, 1849, **1**, 244. He gave a general account of his work at a Friday evening discourse at the Royal Institution on April 27th, 1849, which was published as a brochure entitled, "Benzole: its Nature and Utility."

firm in Huddersfield. The fatal accident occurred in a laboratory in the east part of London on February 17th, 1855.*

The total number of definite compounds actually known or suspected to be contained in coal-tar at the time of Mansfield's work was thirteen. Of these four were only conjectured to be present, and one, as we know, had been wrongly identified with cumene. What Mansfield did was to show conclusively that benzene could be obtained if required in any quantity from coal-tar, "naphtha," that toluene was also a constituent of this naphtha, and that the higher homologues were there if wanted. There was something prophetic about this statement, which occurs in the introductory portion of his paper:

"It appears somewhat strange that, in this country, where coal-tar is so exceedingly plentiful, our chemists should have been contented with the discovery of naphthaline, and should have allowed others, less fortunate than ourselves in being able to command abundance of this almost national production, to inform us of the existence at our feet of vast quantities of aniline, of paranaphthaline (anthracene), and of other remarkable substances; and it appears, perhaps, no less singular that we should have failed as yet in applying them, when discovered, to the practical uses which they will no doubt some day claim."

Mansfield went further, however, than simply isolating and characterising benzene and toluene. In 1847 he described and patented a process for preparing nitrobenzene by the action of strong nitric acid (1.5 sp. gr.) upon benzene in glass or earthenware spiral tubes or other suitable form of apparatus cooled by immersion in water. Nitrobenzene must have been prepared in some quantity from coal-tar benzene about that time, since Hofmann, in whose work aniline played a very important part, refers to his having made this material by the reduction of nitrobenzene from this source. In his introductory remarks prefacing the volume of Reports of the Royal College of Chemistry (1849), which volume comprises Mansfield's paper, Hofmann says with respect to this:

"Nor is the sense of sight the only one which benzole promises to serve (referring to its use as an illuminant). By treatment with nitric acid the same volatile hydrocarbon yields a fragrant oil, the

* Prof. A. H. Church, F.R.S., informs the writer that Mansfield was then preparing specimens of benzene and its homologues and derivatives for the French International Exhibition. The accounts are not inconsistent; he may have been carrying on both lines of work, or Read Holliday's specimens may have been intended for the Exhibition. Unfortunately, the chief figures in this misfortune have all passed away. An obituary notice was published by the Chemical Society in 1855; *Quart. Journ.*, 8, 110.

odour of which is not to be distinguished from that of oil of bitter almonds; so that this perfume may now be procured from coal-tar in tons, if required, with the greatest facility and at a trifling cost." As a matter of fact, nitrobenzene, under the name of "essence de mirbane," had been introduced into commerce by C. Collas, of Paris, as a substitute for bitter almond oil, and was chiefly used for scenting soap, but this limited application of a tar product, although interesting historically, was practically of no importance from the industrial point of view. According to Bolley (*Handbuch der Chemischen Technologie*, Vol. V., Part II., p. 257, 1870), Collas must be credited with the use of a mixture of nitric and sulphuric acids, the modern process for nitrating benzene, although, for reasons not now obvious, he specifies the use of the "monohydrated" nitric acid.*

The "practical uses" which Mansfield had predicted for the coal-tar hydrocarbons began seriously in 1856 with Perkin's discovery of mauve, and the establishment of the Greenford Green factory in 1857 for the manufacture of this first of the coal-tar colouring matters. It must not be imagined that no use for coal-tar had been found up to that date. Tar distilling as an industry was carried on extensively, but the products were entirely applied to what may be described as coarse uses, such as timber preserving, an industry which had been founded by Bethell in 1838, and which led to a large consumption of the "creosoting" oils. The "naphtha" also was used as a solvent or for burning in lamps, and the pitch for coating surfaces of wood or metal which required protecting from corroding influences. It is interesting from the historical point of view to read in "A Journey through England and Scotland to the Hebrides in 1784," by a distinguished French author, Faujas de Saint Fond, of which a revised translation has recently been given by Sir Archibald Geikie (Glasgow: Hugh Hopkins, 1907), the following statement relating to the use of the crude tar for coating ships:

"The harbour of Leith, when we entered it, was full of vessels, English, Scottish, American, etc. I saw several vessels belonging to Glasgow and Leith which were coated over with bitumen or tar, extracted from pit coal at the manufactories of Lord Dundonald, who has introduced the making and using of this tar on a great scale in England. The vessels covered with it appeared of a fine shining black, which distinguished them from the others. Several ship-masters from the West Indies whom I questioned assured me that their vessels thus tarred arrived in the best possible condi-

* It is possible that he had in mind the old view of an acid as a combination of an "acid oxide" with water, that is, nitric acid as $N_2O_5 + H_2O$.

tion, and were free from worm-holes. Navigation is doubtless much indebted to Lord Dundonald, who has continued with the greatest perseverance to perfect this useful product of coal, and has done everything to bring it into general use in the country—no easy task when it involves the change of old habits.” (Vol. II, pp. 220–221.)

Even at the time of Mansfield's work no coal-tar hydrocarbon had been utilised as a source of other chemical compounds, tinctorial or otherwise, and he himself, in describing the practical applications of benzene, refers only to its use as a solvent or an illuminant. Perkin's discovery thus created a demand for this hydrocarbon as a raw material in a new industry on a scale never before contemplated. Mansfield's experiments had prepared the way, but there had been no demand for benzene, and the tar distillers could not at first supply it in quantity or in a sufficient state of purity. It is of interest to know that the first supply of this material used by Perkin came from the Scotch tar distillery of Messrs. Miller and Co., of Glasgow.

Then came the difficulties connected with the nitration and the reduction of the nitrobenzene to aniline. Here, again, Mansfield had played the part of a pioneer, but his process was impracticable on the scale now required. Moreover, it was too costly, for it must be borne in mind that the new dye had to compete with the existing vegetable colouring matters, and on June 12th, 1856, Messrs. Pullar, of Perth, who had been testing the dyeing properties of mauve, had reported to Perkin that the discovery was a valuable one provided it did not “make the goods too expensive.” It is needless to say that nitric acid of the strength used by Mansfield would have been a very costly material in 1856. In fact, nitric acid of sufficient strength to nitrate benzene could not be obtained in quantity at that period, and Perkin had to devise apparatus for nitrating with a mixture of sulphuric acid and sodium nitrate. His resourcefulness is well revealed by this passage quoted from his Hofmann Memorial Lecture in 1896: “At this time neither I nor my friends had seen the inside of a chemical works, and whatever knowledge I had was obtained from books. This, however, was not so serious a drawback as at first it might appear to be, as the kind of apparatus required, and the character of the operations to be performed, were so entirely different from any in use that there was but little to copy from.

“In commencing this manufacture it was absolutely necessary to proceed tentatively, as most of the operations required new kinds of apparatus to be devised and tried before more could be ordered to carry out the work on any scale.” (Trans., 1896, 69, 606.)

After the manufacture of mauve had been started, the demand for the new dyestuff increased to such an extent that the resources of the Greenford factory were taxed to their utmost, and the assistance of another firm had to be called in for supplying raw materials. That firm was Simpson, Maule, and Nicholson, whose factory was at Locksfields, in the south of London. The Nicholson of the firm was that pupil of Hofmann's already referred to as having been a co-worker with Mansfield, and, under his energetic management, they not only supplied the firm of Perkin and Sons with some of the raw materials required, but later they also entered the colour industry, and in 1865 established the Atlas Works at Hackney Wick, the firm being transferred in 1868 to Messrs. Brooke, Simpson, and Spiller. Mr. William Spiller, formerly of this latter firm, has told the writer that he well remembers the early stages in the manufacture of nitrobenzene by their predecessors at Locksfields, where he was then working in association with the late Mr. E. C. Nicholson. The nitration was carried out in large glass "boltheads" arranged in series, as they had not then discovered that cast-iron vessels could be used. The scale of working was quite small as compared with the modern output from a large nitrating still, and they experienced the difficulty referred to by Perkin of obtaining a supply of pure benzene. The operation also was somewhat capricious, owing to the want of uniformity in the quality of the commercial "benzole," and to the absence of mechanical stirring. The cheapening of the process by the introduction of cast-iron stills with mechanical stirring gear did not take place until some time after the manufacture of mauve had been commenced in 1857. The plant in use was described and figured by Perkin in his Cantor Lectures, delivered before the Society of Arts in 1868, and has since been refigured in many works on technology, as it is practically the same in principle as that now generally in use.*

The next step, the reduction to aniline, had also to be worked out on the manufacturing scale. The laboratory method then generally in use was Zinin's, namely, hydrogen sulphide in presence of ammonia, a process obviously impracticable on the large scale. The use of metals, such as tin or zinc, in combination with acids, would have been both costly and unmanageable. Fortunately, however, Béchamp, in 1854, had found that iron and acetic acid

* A workman, James Underwood, in the employment of Simpson, Maule, and Nicholson, at Locksfields, during the early years of the colour industry, also remembers this manufacture of nitrobenzene in boltheads and the development to cast-iron stills. This last improvement is generally attributed to E. C. Nicholson. A figure of the earliest form of (horizontal) still is given by Perkin in his Cantor Lectures above referred to.

could be used for reducing nitro-compounds, and Perkin, who had been familiarised with this process in Hofmann's laboratory, applied it successfully for the manufacture of aniline.* That this was a task of considerable difficulty can be readily understood by those who are familiar with the violence of such "reducing" processes, unless properly controlled. It is, in fact, known that at first serious attempts were made to extract the minute quantity of aniline contained in the coal-tar oils directly by acid washing—a process which, it is needless to say, had soon to be abandoned on account of its cost and the impure state of the product. In the manufacture of aniline from nitrobenzene, the firm of Simpson, Maule, and Nicholson also co-operated with Perkin and Sons, and Mr. William Spiller has given the writer a graphic description of their early work at Locksfields when starting this branch of the industry. The reduction was carried out in iron vessels with removable still-heads, the vessel being at first uncovered, and the materials, nitrobenzene, iron turnings, and acetic acid, simply stirred up by a rod until the reaction showed signs of starting. The still-head was then immediately clapped on, and a workman mounted guard with water-hose ready to play over the still if the contents gave signs of boiling too violently. The cost of the acetic acid was a considerable item at that time, and they had to make their own acid by heating sodium acetate with sulphuric acid. It was soon found that hydrochloric acid could be used instead of acetic acid, and the introduction of stills with mechanical stirrers put this branch of the manufacture on a sure basis. It is perhaps hardly necessary to point out that the "aniline" of that period was a mixture of homologues, and very impure from the modern point of view.

And so the manufacture of the first of the "synthetic dyestuffs" was started at Greenford Green towards the end of the year 1857, and the genius of the founder had ample scope for exercise. Let it be borne in mind that the raw product obtained by oxidising crude aniline with sulphuric acid and potassium dichromate was what would now be called a "resinous mess." Processes for its purification had to be devised, and here again the resourcefulness of Perkin becomes manifest. With that true scientific spirit which dominated all his work, the investigation of his products and processes was always kept going. At first the crude product was collected on filters and washed with water to remove excess of aniline sulphate, then dried and powdered, and extracted with coal-tar "naphtha" until free from resinous impurities, then dried

* "Had it not been for this discovery the coal-tar colour industry could not have been started."—W. H. Perkin, Hofmann Memorial Lecture, *loc. cit.*, p. 607.

gain and extracted with methylated spirit, and the filtered solution distilled until the dyestuff separated out. This method of purification was afterwards improved and cheapened by the omission of the naphtha treatment, as it was found that dilute methylated spirit extracted the colouring matter directly, and left the resin undissolved. The process was finally simplified by boiling out the colouring matter with water alone, and precipitating with an alkali so as to obtain the free base, which was then converted into acetate or use by the dyers.

The discovery and manufacture of mauve, with its train of consequences, must be regarded as constituting but a portion of Perkin's claim to our gratitude. In starting upon this work he had, against the advice of his illustrious master, Hofmann, broken away from the path of pure science and entered a field in which he was a novice. His whole future was bound up with the success of the undertaking, for his father had placed nearly his entire capital in the venture in order to establish the factory at Greenford Green. There was evidently something more to be done besides placing the new dyestuff on the market. The dyers and printers had to be convinced of its merits and taught how to use it. This task, by no means a light one, had also to be undertaken by Perkin, who, up to that time, had never been brought into contact with the tinctorial industries. It has frequently been mentioned that Messrs. Pullar, of Perth, were the first to give encouragement to the young inventor so far as concerned the dyeing properties of mauve. At their instigation it was tried for silk dyeing by Thomas Keith, silk dyer, of Bethnal Green, London, and he also reported favourably. But, as is generally the case with new departures, the step from the experimental to the practical scale was not made without encountering difficulties. It was found that on the large scale the dye "took on" unevenly, and caused a patchy appearance, so that a restraining material had to be added to the bath. The use of the soap bath for silk dyeing was the outcome of Perkin's association with a practical dyer, and Keith's dyehouse was the first in which mauve was used on the industrial scale.

Then with respect to wool and cotton dyeing, the same pioneering work had to be done. Perkin has told us that he and Mr. (now Sir) Robert Pullar had independently discovered the use of manganin and a metallic oxide as a mordant for cotton dyeing, and, in conjunction with Alexander Schultz, he had introduced the "insoluble arsenite of alumina" as a mordant. The calico printers in this country did not at first take kindly to the new colouring matter, and Perkin has often told the writer that the impetus to

this most important application of his discovery came from France. It appears that, owing to some technical oversight, the French patent was ineffective, and the French manufacturers accordingly began making the new dyestuff themselves. It was in France, in fact, that the term "mauve" was given. With the well-known skill of the French calico printers, beautiful designs in mauve were produced and sent over to this country, and this was more effective than any other cause in hastening the use of the dye for this purpose over here. Had it not been for this stimulus the success of the new factory would have been doubtful, for Messrs. Pullar had reported to Perkin that, in their opinion, unless the new dye could be used by the printers it would be questionable whether "it would be wise to erect works for the quantity dyers alone will require." * In summing up this part of his experience Perkin stated in 1896:

"Before the aniline purple could be introduced for dyeing woollen and mixed fabrics, some weeks were also spent at Bradford in finding out suitable methods of applying it.

"Thus it will be seen that, in the case of this new colouring matter, not only had the difficulties incident to its manufacture to be grappled with, and the prejudices of the consumer overcome, but, owing to the fact that it belonged to a new class of dyestuffs, a large amount of time had to be devoted to the study of its applications to dyeing, calico printing, etc. It was, in fact, all pioneering work—clearing the road, as it were, for the introduction of all colouring matters which followed, all the processes worked out for dyeing silk, cotton, and wool, and also for calico printing, afterwards proving suitable for magenta, Hofmann Violet, etc." (Hofmann Memorial Lecture, *loc. cit.*, p. 609.)

The success of the new industry had for its natural consequence the creation of a host of imitators. All kinds of oxidising agents were tried upon aniline and made the subjects of rival patents. The departure from the original patent was in some cases so slight that it is questionable whether in modern patent legislation the inventor's claim would not be dismissed as a "colourable imitation." Tabourin and Franc Bros. claimed aniline hydrochloride instead of sulphate; Beale and Kirkham in England, as well as Scheurer-Kestner, Depouilly and Lauth, Coblenz, and C. Phillips in France, claimed bleaching powder; Smith claimed chlorine

* "I distinctly remember the first time I induced a calico printer to make trials of this colour that the only report I obtained was that it was too dear, and it was not until nearly two years afterwards, when French printers put aniline purple into their patterns, that it began to interest English printers."—Perkin's Cantor Lectures, Society of Arts, December 7th, 1868, p. 9.

later, Greville Williams potassium permanganate, Kay manganese peroxide, David Price (attached to the firm of Simpson, Maule, and Nicholson) claimed lead peroxide, Dale and Caro cupric chloride, Stark and Guyot red prussiate of potash, and so forth. It is needless to point out that many of the products obtained by these inventors could not have been Perkin's mauve at all, and, as a matter of fact, not one of these rival processes was enabled to compete successfully with the original "bichromate" method. The field was too small or the colour too difficult to purify, or the oxidising agent too expensive, although at that time the bichromate cost from 10d. to 11d. per pound. The only one of these processes which gave a good result was Dale and Caro's, but even this could not be worked so economically as the original process.

The introduction of mauve by the founder of, and pioneer in, this new development in manufacturing chemistry soon led to the further discovery of coal-tar colouring matters and to the establishment of other factories. For about a decade the manufacturing operations at Greenford were carried on successfully, and without any fresh discovery of very great importance, although Perkin's activity in the field of pure scientific investigation never ceased. Magenta was first made industrially by Verguin, in France, in 1859, and the firm of Simpson, Maule, and Nicholson soon began to manufacture this on the large scale by the arsenic acid process as well as other well-known colouring matters. Such was the development of the industry that, in 1862, the year of the International Exhibition in London, Hofmann gave a Friday evening discourse at the Royal Institution (*Chem. News*, 6, 90), from which it appears that the definite compounds which had been isolated from coal-tar, and which in Mansfield's list of 1848 consisted of thirteen, had then risen to about forty. It was for that Exhibition that Messrs. Simpson, Maule, and Nicholson prepared a crown of magenta crystals (acetate), which Hofmann exhibited during his lecture, the title of which was "Mauve and Magenta." The selling price of the new dyes at that time may be gathered from the circumstance that the purified solid mauve sold for about the same price as platinum, weight for weight, and the vat from which the magenta "crown" had been crystallised contained a weight of the acetate of that base valued at £8,000, the crystals adhering to the wire framework of the crown being valued at £100.* The discovery and manufacture of magenta was undoubtedly,

* Some of the original crystals are now in the possession of Mr. William Spiller. The trade catalogue of the firm of Simpson, Maule, and Nicholson, placed at the printer's disposal by Dr. Cain, shows that in 1866 "Pure Roseine," was priced at 6d. per ounce.

after the production of mauve, the most important contribution to the industry made during the decade referred to. This discovery did not at first affect Perkin's operations; mauve still held its own, and in 1859 Perkin's brother Thomas, the business man of the establishment, patented on behalf of the firm a process for making magenta by oxidising crude aniline with mercuric nitrate.* This was an improvement upon the original stannic chloride process of Verguin, but it was dangerous, capricious, and expensive, and was very soon displaced by Medlock's arsenic acid process worked by Simpson, Maule, and Nicholson, and also, as the result of a celebrated lawsuit, by Messrs. Read Holliday and Sons, of Huddersfield. But although Perkin and Sons never made magenta in any quantity, the introduction of this dyestuff led to new and necessary developments in their factory. About five years after the foundation of the Greenford works, Hofmann, who had then enthusiastically entered the field of colour chemistry, found that magenta when ethylated or methylated gave rise to violet colouring matters, the manufacture of which was at once taken up by Simpson, Maule, and Nicholson.† Hofmann's Violets and certain phenylated rosanilines, discovered about the same time by Girard and De Laire, in France, and made here also by Simpson, Maule, and Nicholson, soon began to enter into competition with mauve.

It has not, I think, been sufficiently dwelt upon by any of the historians of the coal-tar colour industry that Perkin's pioneering discovery reacted upon itself, for there can be no doubt that the production of aniline on the large scale led to the discovery of processes for the manufacture of magenta, and it was the derivatives of the latter that first began seriously to displace mauve. The discovery by Lauth of colouring matters, such as methyl-violet, formed by the oxidation of the alkylated anilines and manufactured in France about 1866, brought into the field other competitors with the original mauve. The newer dyes were not so fast as mauve, but they were much more brilliant, and fastness soon gave way to brightness. The practical effect of these later developments made itself felt in the gradual decline in the demand for mauve, the use of which soon became very limited, and finally

* "Das Zinnchlorid wird durch das Quecksilbernitrat ersetzt, mit dem die Fabrikation auch in Deutschland ihre ersten, kräftigen Wurzeln fasst."—H. Caro, *Ber.*, 1892, 25, 1031.

† The manufacture of methyl and ethyl iodide on the large scale was a remarkable achievement at the time. When the writer entered the Atlas Works, in 1877, the Hofmann Violets were still being manufactured, and the use of these colouring matters by English dyers continued for more than twenty years after that date. The violet is priced in the 1866 catalogue of Simpson, Maule, and Nicholson at 3s. per ounce.

died out altogether. As a flourishing branch of the colour industry it may be said that mauve did not complete ten years of its existence. But Perkin was enabled to keep the Greenford works going successfully in spite of the adverse influence of the new discoveries and the coming into existence of other factories. He introduced, in 1864, a very ingenious method for the indirect alkylation of magenta, which enabled their firm to compete with the other violet colouring matters then in the market. This method consisted in heating magenta base with methylated spirit—afterwards improved by substituting methyl alcohol—and the compound formed from turpentine oil and bromine in the presence of water. This "brominated turpentine" had long been known to chemists, and had been investigated by Greville Williams, but had never before been used for manufacturing purposes. The dyes thus made were introduced under the name of Britannia Violet of different shades of blueness, according to the degree of alkylation. It was at first thought that they contained the terpene radicle, although it was afterwards considered that they were of the same type if not identical with the Hofmann Violets, so that Perkin had really discovered an indirect method of methylation of a type unknown in chemistry at that time. Perkin's process was very successful, although they were handicapped by having to purchase magenta base, which they did not themselves manufacture. But, on the other hand, brominated turpentine was cheaper as an alkylating agent than the methyl iodide used in the manufacture of Hofmann Violets.

After eleven years' successful working at the Greenford Green factory with mauve and certain of its derivatives, the Britannia Violets, and a few other dyes which are given in the list on p. 2253, a new impetus suddenly came through the announcement, in 1868, that Graebe and Liebermann, in Germany, had discovered that alizarin, the colouring matter of the madder plant, was a derivative of the coal-tar hydrocarbon, anthracene, and not, as had formerly been supposed, a derivative of naphthalene. The German chemists, both of whom are happily still with us, found also that the compound could be prepared from anthracene, and thus was accomplished the first laboratory synthesis of a natural colouring matter.

The demand for another coal-tar hydrocarbon, anthracene, in large quantities and in a state of purity, necessitated further pioneering work. Supplies of the crude material had to be procured, the tar distillers had to be educated in the production of raw anthracene, and factory methods of purification had to be devised. All these requirements were met by the science and

skill of Perkin, then a young man just turned thirty years of age. The subsequent development of the artificial alizarin industry is too well known to need recapitulation in this notice. But there is one point in connexion with Perkin's work in this field which must not be forgotten, and that is the great importance of the dichloroanthracene process in this country at the outset of the new branch of the coal-tar colour industry.

The two processes discovered by Perkin were the anthraquinone process and the dichloroanthracene process. In the first of these the anthracene is oxidised to anthraquinone, the latter sulphonated by heating with strong sulphuric acid to a high temperature, and the sodium sulphonate converted into alizarin by alkaline fusion. The sulphonation by this process yields a mixture of mono- and di-sulphonic acids, and the final product is therefore a mixture consisting of alizarin, anthrapurpurin, and some flavopurpurin. This was the process first tried on the large scale by Perkin, as well as by the German manufacturers. The second process, which was patented here by Perkin a few months after the patenting of the anthraquinone process, namely, in November, 1869, sets out from dichloroanthracene, which is sulphonated by ordinary strong sulphuric acid and the product submitted to alkaline fusion as before. Now dichloroanthracene sulphonates more readily than anthraquinone, and as the product consists chiefly of a disulphonic acid of anthraquinone, the "artificial alizarin" obtained by this process consists mainly of anthrapurpurin with some alizarin and flavopurpurin. Alizarin gives bluer shades of colour than anthrapurpurin, so that although for certain purposes where bright red was required the mixture obtained by Perkin's second process possessed an advantage, for the production of the bluer reds the anthraquinone product had the advantage. Perkin met this difficulty to some extent by devising a method for separating his "alizarin" into "blue shade" and "scarlet shade," but this method was not easy to carry out on the large scale, and added to the cost of the final products.

For the first few years the Badische Company, which had acquired the Caro-Gräbe-Liebermann patent, worked by mutual arrangement in combination with the Greenford Green factory, the latter having the monopoly of the English markets.* The Germans were using the anthraquinone process almost exclusively, this being the method still in use. When ordinary English oil of vitriol is used for sulphonating, a great excess of acid is necessary, and there is much loss owing to the high temperature, so

* The amicable arrangement between the German and English manufacturers was brought about through the mediation of Dr. Hugo Müller, F.R.S.

that the dichloroanthracene process from this point of view had the advantage. Moreover, when anthrapurpurin was the main object of manufacture, it was found that the product obtained by the dichloroanthracene process gave much purer shades than that obtained by the anthraquinone process.* It would have naturally occurred to Perkin in working out this last process to try fuming sulphuric acid as a sulphonating agent, and he did so with success, but this method, although giving better results in the way of yield and uniformity of product, was placed at a disadvantage here on account of the cost of the fuming acid. The advantages arising from this method of sulphonating are an increased yield on account of the lower temperature at which the acid does its work, and a product which consists mainly of the monosulphonic acid, and which therefore gives chiefly the true "alizarin" on alkaline fusion. Now Germany was, at that time, the only country in which the manufacture of fuming sulphuric acid was carried on, and this gave them a distinct advantage in working the anthraquinone process. Perkin has called attention more than once to the state of affairs in this country during the early life of the artificial alizarin industry, and his own statements may be quoted here:

"On account of the expense and difficulty in getting Nordhausen sulphuric acid imported into this country—few vessels liking it as a cargo—we commenced working with ordinary sulphuric acid. We usually employed four or five parts of this to each part of anthraquinone and heated the mixture to $270-280^{\circ}\text{C}$ I find we employed this process principally in our works until the middle of June, 1870. We then began to work on a larger scale than we had hitherto done with dichloroanthracene, and carried both processes on for a time, but finding the latter the most economical, partially on account of the ease with which it yielded the sulpho-acids with ordinary sulphuric acid, we employed it almost exclusively after a time, although frequently making colouring matter by the other method.

"The large quantity of ordinary sulphuric acid which had to be employed to convert anthraquinone into the sulpho-acids, and the high temperature which had to be used, causing a certain

* Perkin, *The History of Alizarin, &c.*, 1879, p. 26. Dr. Caro informs the writer that since 1870 the Badische Co. employed also the dichloroanthracene process for the manufacture of a special kind of "alizarin," consisting chiefly of anthrapurpurin. It may be pointed out, also, that, owing to some peculiarity in the internal administration of the German Patent Laws at that time, the rights of Caro, Graebe, and Liebermann could not be secured in certain States, and so other manufacturers took up the artificial alizarin industry and entered into competition with the Badische Co. So far as the writer has been able to learn, the anthraquinone process was generally employed.

amount of destruction to take place, evidently showed that it was desirable to employ fuming sulphuric acid in this process. In this country we found it costly, but as it was more readily procurable in Germany, the manufacturers there used it. They were afterwards supplied with a very strong fuming acid from Bohemia, containing about 40 per cent. of sulphuric anhydride." (*The History of Alizarin*, etc., 1879, pp. 24-25.*)

The same statement was repeated in substantially identical terms in 1896. Referring to the loss of anthraquinone when ordinary sulphuric acid is used, he says: "The means of overcoming this difficulty was to use fuming sulphuric acid, with which anthraquinone combined at a much lower temperature, but the only acid of the kind then made was the old-fashioned Nordhausen acid. We imported a quantity of this, and, of course, found it to work satisfactorily, but the difficulties and expense connected with the carriage and transport of this substance on account of its dangerous nature—supplied as it then was in large earthenware bottles—made it unsuitable for use in this country.

"The artificial alizarin we first made was produced by the anthraquinone process, the method still used for its manufacture, but the difficulty in preparing the sulphonie acid in those early days just referred to caused us to turn our attention to the second process I had discovered, in which dichloroanthracene was used. . . . Without this process the manufacture of artificial alizarin in this country could not have been carried on with much success in the early days of its manufacture." (Hofmann Memorial Lecture, *loc. cit.*, p. 631.)

The "contact," or "catalytic," process for producing sulphuric anhydride, introduced about the same time in this country by Messrs. Chapman, Messel and Co., and in Germany by the late C. Winkler, dates from 1875, so that Perkin's share in the founding of this great industry does not consist only in his having given us the practical methods for realising Graebe and Liebermann's synthesis in the factory, but in having devised a process which, so to speak, enabled the new industry to be nursed through its infancy in this country and without which it would probably not have survived that Continental competition which, as Perkin has told us, first began to make itself seriously felt about the end of 1873 (*History of Alizarin*, etc., 1879, p. 31). By that time it was fully realised that a complete revision of the

* The use of Nordhausen acid for the anthraquinone process in Germany began about 1871; the introduction of the stronger acid referred to by Perkin in the above passage is generally attributed to Koch in 1873. Dr. Caro informs the writer that he has been unable to find the authority for this statement.

plant at Greenford Green had become necessary. It required enlarging and modifying in order to meet the successful competition arising from the development of the anthraquinone process in Germany, and a considerable expenditure of capital would have been necessary to carry out this work. But Perkin, whose ambition it had always been to be able to devote himself to pure science, and whose personal requirements were extremely modest, found that his manufacturing career had by then provided him with sufficient means to enable him to retire, and, rather than incur the responsibility of making a fresh start, he took advantage of the opportunity for withdrawing altogether from the industry. His career as a manufacturer terminated in 1874, the Greenford Green works having then been purchased by Messrs. Brooke, Simpson, and Spiller, which firm, soon afterwards, transferred them to Messrs. Burt, Bolton, and Haywood, who shifted the manufacture from Greenford Green to Silvertown, and ultimately from this firm the "British Alizarine Company" was developed, and is still at work. Perkin always wished it to be known that he considered the Silvertown works as the lineal descendant of the first coal-tar colour factory.

This sketch of the founding of the coal-tar colour industry is necessarily limited to the history of the Greenford Green factory. These works would now appear quite insignificant in comparison with one of the great German establishments, and the whole output of dyes during the seventeen years that Perkin was connected with them was not very great as measured by modern standards. Nevertheless, it may fairly be said that no single factory established in this country has ever given rise to such world-wide developments, both scientific and industrial. When it fell to the writer's lot to take part in the organisation of the jubilee celebration of 1906, it appeared desirable to place upon record the complete history of the Greenford Green factory as a colour-making establishment, and Sir Wm. Perkin was good enough to prepare the following list:

THE PRODUCTS MANUFACTURED AT GREENFORD GREEN, 1857-1873.

Mauve.—Large quantities manufactured.

Dahlia.—Ethylmauveine, $C_{27}H_{28}(C_2H_5)N_4, HCl$. Made about the same time as Hofmann's Violet [1863]. The colour was much admired, but being very expensive was not largely used (*Journ. Chem. Soc.*, 1879, 35, 399).

Aniline Pink.—First found in washings from mauve, afterwards produced by oxidising mauve with lead peroxide. It is para-

safranine. Made about the same time as Dahlia (*Journ. Chem. Soc.*, 1879, **35**, 407). The researches were made many years before publication.

Magenta.—Prepared by a mercuric nitrate under a patent in my name; a communication from abroad. It was first obtained in crystals in this way (*Journ. Chem. Soc.*, 1862, **15**, 238-240. The research was made some years before publication). The process was dangerous, and not carried on very long.

Amidoazonaphthalene.—Used in a finely precipitated form as an orange, red, or scarlet pigment for calico printing, but not largely.

Britannia Violet (various shades).—Made from Magenta, the bromine compound of turpentine, and methylated spirit, or, better, purified wood spirit. At first thought to be a turpentine derivative, but afterwards found to be methylated rosanilines. Made in large quantities.

Perkin's Green.—This was an interesting compound made by treating Britannia Violet (blue shade) with acetyl chloride. The latter was made in large quantities from phosphorus trichloride and acetic acid. The phosphorus trichloride was made in cast-iron retorts with iron condensers from phosphorus and dry chlorine. The colouring matter was obtained in a crystalline condition, but was not investigated as to its constitution. It was rather extensively used for calico printing when Iodine Green was too expensive.

"Alizarine".—Produced very largely, chiefly by dichloroanthracene process. It consisted of anthrapurpurin and alizarin, chiefly of the former. These were also separated and sold as "Blue Shade Alizarine" and "Scarlet Shade," but we chiefly sold the mixture known as "Red Shade." Besides the above we made suitable mixtures of aniline salts, oxidising agents, and copper compounds for the production of Aniline Black on the fabric by calico printers. Also the colouring matters were made into "lakes" by processes of our own for paperhangings and lithographic and other printing inks in considerable quantities.

This list contains what may be regarded as Perkin's direct contribution to the colour industry as a manufacturer. It may not appear very imposing to us now, but we must read into it all that it means in order to appreciate its full significance. There must be taken into consideration the pioneering work in every direction that had to be done in order to accomplish these results. It must further be remembered that they were achieved at the outset by a youth of about 18, and brought to a successful termination in seventeen years by a young man 36 years of age, and that during the whole of that period, while the factory was actively at work,

a continuous stream of scientific research was kept going in his laboratory. The stupendous consequences of the initiation of this industry must also be borne in mind, and then the extent of our indebtedness to him will be fully realised.

By many who regard manufacturing industry from a narrow point of view, Perkin, as already stated in the previous part of this notice, has been censured for withdrawing so soon from the scene of his industrial operations. The reply to this charge is obvious. He had made a sufficient fortune for his modest requirements, and the seeds which he had sown were developing rapidly in this country. At that time (1874) German competition was only just beginning to make itself felt. The industry was flourishing here, and with respect to France it may be said that within a very short period of the founding of the Greenford Green factory, and especially from the time of the discovery of magenta, the industry was also in a prosperous condition. How thoroughly this branch of manufacture had its head centre in England during the few years following the opening of the Greenford works may be inferred from the fact that such men as Maule and (especially) E. C. Nicholson, both pupils of Hofmann's, had entered the industry; that in Manchester the firm of Roberts, Dale and Co. had secured the services of men like Caro and Martius, who later became pioneers in the German colour-making industry. Or, if we turn to the actual products, we find that in addition to those emanating from the firm of Perkin and Sons, Simpson, Maule, and Nicholson had secured the first really valuable process for making magenta, namely, the arsenic acid process of Medlock; that they had also secured the beautiful process of Girard and De Laire for phenylating magenta so as to convert it into blue and violet colouring matters, and that Nicholson, by his discovery of the method of sulphonation, had developed these into what were for many years the most important of all the coal-tar colouring matters. This firm had also introduced aniline-yellow (aminoazobenzene), the precursor of the basic azo-dyes, and phosphine (chrysaniline),* the first member of the acridine series. They were, moreover, the only manufacturers of the alkylated rosanilines under Hofmann's patent. Then the firm of Roberts, Dale and Co. were making picric acid, and had, through Caro, given to the industry the first induline obtained from aniline-yellow and aniline, as well as Manchester brown or Bismarck brown. This firm had also, through Martius,

* In the 1866 catalogue of this firm, already referred to, Aniline-yellow is priced at 2s. and "Phosphine" at 3s. per ounce. The Nicholson Blues were, at that time, sold only in solution, the price ranging, according to the brand, from 15s. to 30s. per gallon. Solid "Regina Purple" is priced at 15s. per ounce.

given us the dinitronaphthol known as Manchester yellow. Cyanine, or quinoline blue, the first representative of a group of colouring matters which have since become of great importance as special sensitisers for photographic purposes, was discovered the same year as mauve (1856) by Greville Williams, who was for some time chemist at the Perkin's factory, and who afterwards, with Messrs. E. Thomas and J. Dower, started the Star Chemical Works at Brentford. This country may also claim to have been the pioneer, through Crace-Calvert and Lowe, of Manchester, in the technical production of highly purified phenol.* The first successful method for printing on the fabric with aniline-black was discovered and patented in 1863 by John Lightfoot, of Accrington.

This was the state of affairs during Perkin's connexion with the industry, and, superadded to this manufacturing activity, was the supremely important fact that, until 1865, the great master, Hofmann, was among us, and that his laboratory at the Royal College of Chemistry had become a centre of active research in the chemistry of colouring matters which stimulated the industry and supplied chemists for the factories.† Nor must it be forgotten that Peter Griess, the founder of diazo-chemistry, was working over here during the greater part of the same period. It cannot be said that Perkin abandoned the ship in a sinking condition; on the contrary, she was steaming full speed ahead! For any scuttling that may have afterwards occurred he can in no way be held responsible.

ADDENDUM.

As the introduction of fuming sulphuric acid played such an important part in the early history of the artificial alizarin industry, it is of interest to append the following account kindly furnished by Hofrath Dr. Caro. It may be pointed out that the "contact" process for producing sulphuric acid dates from 1875;‡ and therefore subsequently to Perkin's retirement, so that it was

* The state of the industry here and in France five years after its inauguration at Greenford Green can be ascertained from Hofmann's report on the chemical exhibits at the International (London) Exhibition of 1862. It is not going too far to say that during its early years the coal-tar colour industry was essentially English and French.

† Hofmann left London in 1865. From that time until the creation of the Chair of Organic Chemistry at Owens College, Manchester, in 1874, to which Schorlemmer was appointed, there was no Professorship in this department of the science in this country.

‡ The patent of Messrs. Chapman and Messel is dated September 18th, 1875. Winkler's process was described in *Dingler's Polytechnisches Journal* for October, 1875. Dr. Messel gave a description of their process before the Chemical Society in April, 1876, but the paper was not published by the Society.

his successors who had the advantage of this new branch of manufacture:

"Previously to the publication of Clemens Winkler, the entire 'Nordhausen Fuming Sulphuric Acid' was manufactured by John David Starck in Bohemia (in several works near Pilsen), and was largely imported into England. It originally contained about 20 per cent. of the free anhydride. This acid was employed by Perkin in his first experimental manufacture in 1869 for sulphonating anthraquinone, and was afterwards in 1870 exchanged for ordinary sulphuric acid,* while we (the Badische Co.) commenced at this same period with the ordinary acid and gradually went on increasing its strength by adding fuming acid containing about 24 per cent. of free anhydride. I recollect that in 1873 we used chiefly a mixture of two parts of the said fuming acid with one part of the monohydrate. At the same time we studied carefully the effect of the increased strength of the sulphonating agent upon the separate production of the mono- and di-sulpho-acids of anthraquinone, and I believe that at the same time (1873) similar experiments were made by all German alizarin makers, particularly by Gebrüder Gessert and Co., at Elberfeld, and that in consequence of the superior results obtained by the action of stronger acid at a corresponding lower temperature a demand was created for fuming sulphuric acid of greater strengths than hitherto supplied. Thus John David Starck was led to manufacture the solid fuming sulphuric acid containing about 45 per cent. of the free anhydride. This was, I think, in 1873 or 1874. In 1875 we employed regularly the fuming acid of 45—50 per cent. of anhydride. In 1877 we went further in increasing the energy of the sulphonating action by the employment of fuming acid of from 68 to 72 per cent. of free anhydride, which we prepared by distilling the anhydride from one portion of fuming acid into another portion of fuming acid containing 45—50 per cent. of free anhydride. We also distilled the anhydride into the sulphonating mixture of anthraquinone with fuming acid. Immediately after the publication of Winkler, in 1875, we commenced experimenting with his synthetical process, and after having many times changed our experimental plant, we succeeded in manufacturing the fuming acid on a very large scale from 1877. At about the same time other manufacturers started the manufacture of fuming acid by the synthetical process."

R. MELDOLA.

* See Perkin's statement (*ante*) quoted from his *History of Alizarin*, 1879.

ROBERT WARINGTON.

BORN AUGUST 22ND, 1838; DIED MARCH 20TH, 1907.

THE name of Robert Warington will ever be associated with one of the most important advances in the agricultural chemistry of the latter half of the nineteenth century, although his classical work on nitrification, which may be regarded as his life-work, bears but a small proportion to the total of that accomplished by him. He, no doubt, owed his chemical proclivities to his father—a Robert Warington also—who was a prominent figure amongst the chemists of earlier days. The elder Warington was one of the first chemical assistants at University College, and was subsequently appointed chemical operator to the Society of Apothecaries. He also was a Fellow of the Royal Society, and published several papers on chemical subjects; yet chemistry is more indebted to him for the part which he took in founding the Chemical Society than for the extent of his own original work. It was through his zeal and powers of organisation that this Society was founded in 1841, and the work which he did for it as its secretary during the subsequent ten years helped in no small measure to launch it on its prosperous career.

Robert, his eldest son, was born on August 22nd, 1838, in the parish of Spitalfields. His mother was a daughter of George Jackson, M.R.C.S., to whom science is indebted for several improvements in microscopes which have not yet been superseded, as well as for the invaluable ruled glass micrometer. The original dividing machine made by him for ruling the lines was still being used by a well-known optician in 1899, and is probably in use at the present time.

Very early in young Warington's life his parents took up their residence at the Apothecaries' Hall, and it was here, in the uncongenial atmosphere of the City, that he spent his childhood and youth. His constitution was naturally feeble, and a life in the heart of London, with but little exercise, and no companions of his own age to assort with, did not tend to strengthen it. All through life he had to contend with a lack of bodily vigour, which rendered his work doubly laborious to him. For his education he seems to have been chiefly indebted to his parents. While still quite young he studied chemistry in his father's laboratory, and had the advantage of attending lectures by Faraday, Brande and Hofmann.

In consequence of the unsatisfactory state of young Warington's health, his father sought to get him some employment in the country, and, with that object in view, applied to Mr. Lawes, with

whom he was acquainted, and for whom he had done some professional work. The outcome of this was that in January, 1859, the youth went to work in the Rothamsted Laboratory as Lawes' unpaid assistant. Here he remained for one year, devoting all his time to ash analyses, of which he had had no previous experience, and examining various methods for obtaining the most satisfactory results. Dr. Pugh and Mr. F. R. Segeleke were also working in the laboratory at that time, and they gave Warington valuable assistance in his work. Of the two series of analyses eventually completed, the first comprised those of the ashes of grass grown under different manurial treatment, the results of which were published in Lawes and Gilbert's "Report of Experiments with Different Manures on Permanent Meadow Land" (*J. Roy. Agric. Soc.*, 1859, 20, 407), the second series was that of the ash of grain from Broadbalk Field. These latter analyses were never published, their place having been taken by more complete work on the same subject by Richter.

Although Warington left the Rothamsted Laboratory in January, 1860, his interest in the work there never ceased, and, until he resumed his connexion with Lawes a few years later, he devoted much of his time to studying the Rothamsted results, and was a frequent visitor to the laboratory.

His health having been somewhat re-established by his year's residence in the country, he returned to town, and continued to reside with his parents until 1862, spending his days at South Kensington, where he worked under Dr. Frankland as research assistant. But at the end of this period a further breakdown in health forced him again to seek a country life, and he betook himself to the Royal Agricultural College at Cirencester. Here he remained for four and a-half years, the first nine months of which were spent in doing analyses for Dr. A. Voelcker, and the remainder of the time in fulfilling the duties of teaching assistant under Professor Church.

It was during his residence at Cirencester that Warington published the first papers on scientific subjects which appear under his name. These were printed in the *Journal of the Chemical Society*. The earliest of them (1863) dealt with the quantitative determination of phosphoric acid. This was followed by two other short communications on kindred subjects, which preceded and prepared the way for his first work of importance—an investigation into the part played by ferric oxide and alumina in decomposing soluble phosphates and other salts, and retaining them in the soil. The results of this investigation are embodied in a series of four papers read before the Chemical Society, and are typical examples

of the careful work and close reasoning which characterised all Warington's researches. That ferric oxide acted as a fixing agent for soluble substances applied to a soil was already known, but the action was attributed to an indefinable physical attraction, which explained nothing. Warington proved, first by experiments with pure ferric oxide, and then with ordinary soil, that the action in the case of calcium phosphate was simply one of chemical decomposition, resulting in the formation of ferric phosphate, whilst in the case of other salts, such as carbonates, sulphates, nitrates, etc., the chemical character of the action was indicated by the fact that the iron did not retain the salt as a whole, but partially decomposed it, retaining the basic portion in excess over the acid portion.

Warington did not allow his work at Cirencester to sever his connexion with Rothamsted, and he offered to analyse three of the most important of the animal ashes which had been prepared there, on the condition that he might make use of the results thus obtained. He consequently received mixed ashes representing the whole bodies of a fat ox, a fat sheep, and a fat pig, and an abstract of the analyses made by him appeared in an article which he wrote for the second supplement to "Watts's Dictionary of Chemistry." The analyses, together with others by Richter, were also published by Lawes and Gilbert in the *Phil. Trans.*, 1883.

In 1864 Warington commenced lecturing to the students at Cirencester on the Rothamsted experiments, and went systematically through all the work which had already been published, together with many additions of as yet unpublished results which had been communicated to him by Lawes and Gilbert. A desire was expressed at Cirencester that these lectures should be published, and negotiations to that end were, consequently, opened with Lawes and Gilbert. The outcome of these was that Warington was to write a book on the Rothamsted investigations, Lawes guaranteeing him from pecuniary loss, but offering no remuneration. Lawes also reserved to himself the right to supply a preface to the book, on the ground that there would be previously unpublished matter incorporated therein. The writing of this book involved a large amount of labour, especially as, in studying the effect of manures in different seasons, Warington was led to recognise the almost paramount influence of the rainfall on the results, and its action in washing the nitrates out of the soil, an action up to that time unrecognised. For the purpose of examining this action more closely, he compared the results from the plots at Rothamsted with the temperatures and rainfalls supplied to him by Glaisher; at the same time he applied to Gilbert to furnish him with unpublished

data respecting the Rothamsted hay crops. Gilbert, however, objected to what now appeared to him in the light of a publication of Rothamsted results by others than Lawes and himself. Discussions ensued, the upshot of which was that the book remained in manuscript, and the seeds of an unfortunate dissension between Gilbert and Warington were sown. Some 120 pages of this book were written (and are still in existence), but Warington declined the pecuniary compensation which Lawes offered to him for his labour.

Leaving Cirencester in June, 1867, he became chemist to Lawes's manure and tartaric and citric acid factories at Millwall, where he remained until 1876. During these years he generally had a long conversation every week with Lawes on those problems in agricultural chemistry which happened to be under investigation at the time, and which were evidently more congenial subjects of discussion to both of them than the problems arising in the factory. Even these, however, were by no means lacking in interest, and at the conclusion of his engagement at Millwall in 1874, Warington remained in the laboratory there for two years longer, working on citric and tartaric acids, and ultimately publishing his results in a paper of 70 pages in the *Journal of the Chemical Society*. This paper was published with Lawes's approval, and it is noteworthy for the opinion expressed therein, that "the large amount of information acquired in the laboratories of our great manufacturing concerns might well be published without any injury to the individual manufacturer." Eighteen years later, when Warington had for a second time gone to work in Lawes's tartaric and citric acid factory, he published another paper dealing with these acids, and with the detection of the presence of lead in them. With this solitary exception, all Warington's subsequent work was on agricultural chemistry, and all of it was done in the Rothamsted laboratory.

While still at Millwall he had been writing a good deal on agricultural subjects—several articles for "Watts's Dictionary" and for the Agricultural and Horticultural Co-operation Association—and he had, moreover, as already mentioned, been in continual consultation with Lawes as to the Rothamsted results; he was naturally, therefore, prepared to receive Lawes's suggestion that he should go and work in the Rothamsted laboratory. The terms were all settled, and had readily been assented to by Warington; for, although they had involved a reduction of salary to two-thirds of that which he had been receiving at Millwall, he obtained a certain amount of freedom by way of compensation. He was to be at liberty to publish his own work in his own name, provided

that it made its appearance as Rothamsted work; but in cases where the work dealt with subjects which had already occupied the Rothamsted investigators, it was to be published in the joint names of Lawes, Gilbert and Warington. This arrangement, however, owing to some unforeseen difficulties, was not carried out; and it was not until after a delay of two years that Warington went to Rothamsted (in 1876), under an agreement for a year only, to work simply as Lawes's private assistant. The engagement was subsequently extended, and all his results were published, either in his own name or in the names of Lawes, Gilbert and Warington.

Before removing to Harpenden he went to work at the laboratory at South Kensington in order to learn water and gas analysis under Frankland's assistant, some of the Rothamsted soils being sent to him for practising determinations of nitrogen. While there he devised a method of extracting soils by the vacuum pump, which method has since been largely used at Rothamsted. In the autumn of the same year (1876) he made a short tour among the German experimental stations, and then took up his residence for good at Harpenden.

The construction of a gas analysis apparatus (under Frankland's direction) for the Rothamsted laboratory, occupied a considerable time, and, pending its completion, Warington made a study of the indigo method of determining nitric acid. This method, as generally used, he found to be full of sources of error. The principal of these he succeeded in correcting, and, with the method of determination thus rendered trustworthy, he proceeded to determine regularly the nitrates in the drainage-water from the various wheat plots in Broadbalk field. The chlorides were determined at the same time. No such systematic work had been previously done, whilst the methods of sampling which had been adopted when any analysis had to be made had been faulty. Warington now altered these methods, so that the samples analysed should faithfully represent the average composition of the drainage-waters.

Having examined the indigo method for determining nitric acid, he next examined the Crum-Frankland method by agitation with mercury, and subsequently the method of Schloësing, modified, however, in such a way that the nitric oxide produced was determined by gas analysis. The exhaustive examination of these methods of analysis are described in a series of papers published in his own name in the *Journal of the Chemical Society* and elsewhere, extending down to 1882. The modified Schloësing method was the one which he finally adopted, and with it he began a long

series of determinations of nitrates in soils, and in mangels, swedes and potatoes.

Having satisfied himself as to the methods of nitrogen determination, he next turned his attention to those for the estimation of carbon, and having examined the permanganate and the dichromate methods, and found them wanting, he finally adopted the combustion method, which proved to be thoroughly satisfactory, provided that carbonates were entirely removed by prolonged treatment with sulphurous acid. In this work he was assisted by Mr. W. A. Peake, and the results were brought before the Chemical Society in the names of Warrington and Peake.

Warrington's results from the examination of the rain and drainage water, together with results previously obtained at Rothamsted, formed the subject of a very long report published in the names of the three investigators in the *Journal of the Royal Agricultural Society* for 1882. The subject, however, continued to occupy Warrington's attention long after this date, and we find a report on the subject in the three joint names in 1883, and papers by Warrington alone in 1889 and 1887. The last-mentioned paper is an important contribution (*Trans.*, 1887, 51, 500) to the study of well-waters, and deals with the wells in the chalk formation on which Harpenden is situated. In later years (1904) Warrington was enabled to give these results a practical bearing on the supposed contamination of the Harpenden water supply, and he saved the community, at any rate, for a time, from adopting an expensive and, apparently, quite unnecessary system of sewerage.

So far Warrington's work, as here described, consisted largely of examining and perfecting methods of analysis for use in agricultural research. For this work the precision of his nature, and the carefulness of his manipulation, pre-eminently fitted him, and most of the methods of analysis which he elaborated have been accepted as standard methods, which promise to remain in use for many years to come. The remainder of his work, however, is that by which he made his name, and if a strictly chronological sequence of events had been followed it should have been mentioned earlier in this notice, for it was in 1877 that he began to study nitrification, and this subject occupied the foremost place in his mind until 1891, when his opportunities for pursuing the subject ceased. During this period he published about ten papers on the subject, all in his own name, the principal of which were four communications to the Chemical Society, bearing the title "On Nitrification," Parts I to IV.

That the natural conversion of ammonia into nitric acid was

the work of an organism had been suggested by A. Müller as early as 1873, but it had been reserved for Schloesing and Müntz to establish definitely that this was the case. In 1877 they showed that, when sewage was allowed to percolate through a column of sand and limestone, the nitrification which occurred during its passage could be prevented by the presence of a sterilising agent, such as chloroform vapour, and after such sterilisation, the activity of the sand could be resuscitated by inoculating it with a few particles of vegetable mould. Questions affecting the problems connected with nitrogen in the soil had naturally been amongst those to which the Rothamsted investigators had, from the first, devoted themselves, and, consequently, they at once set to work to examine such an important observation as that of Schloesing and Müntz. A complete verification of it was obtained by Warington, operating with garden soil only, and using a solution of ammonium chloride instead of sewage; and he was enabled to add the additional information that nitrification occurred only in the dark. This paper appeared within a year of that of Schloesing and Müntz. Two and a-half years later he published a second paper, which added considerably to the facts already established. He showed that the nitrifying organism, besides requiring darkness in order to do its work, must also be supplied with food for its growth—potash, lime and phosphorus—and, moreover, that all liberation of free acid must be prevented, by the presence of some salifiable base, such as calcium carbonate. He found, also, that after the introduction of a small quantity of active soil or solution into a liquid capable of nitrification, no action occurred until a certain time had elapsed, this period of incubation being probably due to the organisms having to multiply to a certain extent before they become sufficiently numerous to produce recognisable results. An increase of temperature was found to favour the action up to a certain point, and it was shown that various vegetable moulds and known bacteria were not the organisms to which nitrification could be attributed. Many difficulties, however, still remained to be cleared up, notably the want of uniformity of the action, which resulted in the production of nitrates in some instances, and nitrites in others. We now know that the process is performed by two quite distinct organisms, and that their nutrition is, in some respects, wholly different from that of any other organism hitherto studied; but until this knowledge had been gained, work on the subject was singularly difficult, and the results were very perplexing.

Warington's third paper on nitrification added considerably to our knowledge of the circumstances attending the action, and established the fact that the organisms are almost entirely confined

to the first nine inches of ordinary soil. The distribution of the organism in the soil was dealt with still more exhaustively in a subsequent communication in 1887.

The prize coveted by the workers on this subject was, however, the isolation of the organism itself; and to prepare himself for this task, Warington went to London for a time, in 1886, to learn bacteriology under Dr. Klein at the Brown Institution. From Dr. Klein he obtained a large number of pure cultures of various bacteria, and all these, as well as others obtained from his own experiments with soils, he examined as to their behaviour towards ammonia and nitrates, and also as to their mode of growth on skim-milk. The results were brought before the Chemical Society, and proved that none of the bacteria, except the nitrifying organism itself, possessed any appreciable power of nitrification. The majority of the organisms examined, however, were active denitrifiers. Denitrification—whereby nitrates are converted into nitrites, oxides of nitrogen, or even nitrogen gas—was, at this time, a well recognised work of micro-organisms, but was one which naturally enhanced to a considerable extent the difficulties met in elucidating the reverse phenomenon of nitrification. Warington's work added a good deal to our knowledge of the subject, and showed that denitrification is a property actively exhibited by a large number, but by no means by all, micro-organisms, and that in a soil it becomes complete, before the nitrifying organisms begin their task of reversing the reaction. An excellent account of the denitrification of farmyard manure was subsequently written for the *Journal of the Royal Agricultural Society* (1897, 8, Part IV):

Warington's work on nitrification was amply sufficient to establish the fact that the oxidation of ammonia in the soil was the work of an organism, but that organism seems to have been isolated first by Schloësing and Müntz in 1879, although the method which they adopted left, at the time, considerable doubt as to its real identity. But even the isolation of this organism did not solve the whole problem: there was still the independent formation of nitrites and nitrates to be accounted for; and it was here that Warington's work was most conducive to a solution of the difficulties, for he succeeded in proving that one organism alone could not be held accountable for the various phenomena observed, and that two different organisms must be concerned in the process of nitrification. His success all lay in the chemical aspects of the subject. He was the first to obtain (1879) liquid cultures which converted ammonia into a nitrite, and preserved this power in all sub-cultures, but which was incapable of producing any nitrate; and shortly afterwards (1881) he obtained cultures which were able to convert

nitrites into nitrates, but were unable to oxidise ammonia. This was a practical separation of two distinct organisms, but at the time Warington did not grasp the true meaning of his results, and he associated the change from nitrites into nitrates with a white growth which appeared floating in the liquid, but which really had nothing to do with it.

In 1890, after the work of others had resulted in the isolation of the nitrous organism (that which converted ammonia into nitrites), Warington returned to the subject, and found that the white surface organism could not be held accountable for the conversion of the nitrites into nitrates. He eventually succeeded in isolating the organism which really produces this change, and obtained a nearly pure culture of the nitric organism. At the same time he showed that organic carbon is not necessary for the growth of these organisms, as he had previously imagined, but that they can obtain their carbon from carbonates. These results were published in his fourth paper on nitrification (1891), and were communicated to the Chemical Society only a few days before Winogradski made a similar communication to the French Academy. Winogradski, however, had pushed the matter somewhat further, having obtained the organisms in bodily form, and having shown how they could be cultivated on solid media, a problem which had baffled Warington and other investigators. Warington, therefore, had to share his final hard-won success with another.

The practical results of nitrification in the soil were being investigated while the search for the organism was still in progress, and Warington began a long series of determinations of nitrates in the Rothamsted soil, the first results of which were published as a lecture given before the Society of Arts, for which he was awarded a silver medal.

The quarrels even of eminent men are generally better left to bury themselves in oblivion, but we should hardly be doing justice to Warington if we were to pass over in silence the circumstances which made his work so arduous to him, and finally brought it to a premature conclusion. Indeed, there is so much that is pathetic, and even grand, in the unfortunate disagreement which arose and became intensified between Gilbert and Warington, that a brief allusion to the subject cannot lessen our appreciation of either of them. That two of the greatest of England's agricultural chemists should be at variance with each other may afford no subject for wonder, but what must surprise the layman is that in spite of this strong personal disagreement these two should for years continue to work under the same roof, on the same subjects, publish-

ing their results as joint productions. No mere forbearance (of which there was much), no mere love of gain (of which there was none), could have effected this; it was the love of science, pure, simple and unselfish, which could alone accomplish such a task, and obtain a mastery over the more human passions.

When, in 1889, Lawes resigned his active control to the present Committee of Management, it was evident that the work of the station could no longer be carried on in this painful state of tension, and, all attempts at accommodation having failed, the Committee were reluctantly forced to decide that Warington's work there must terminate. This was in June, 1890, and it was arranged that he was to leave in the following January. Having, however, in the meantime, reached a very interesting stage in his work on the nitrifying organism, he petitioned to be allowed to stay on, without remuneration, until June of 1891. This petition was granted, and before that date he succeeded in bringing the work on hand to a successful termination.

Throughout all the trying circumstances of these years Lawes showed an undeviating friendship towards Warington, and Warington's feelings towards Lawes were those of love and veneration. Perhaps, however, the highest tribute which could have been paid to his rectitude and disinterestedness was paid when the Royal Society requested him to undertake the obituary notice of Gilbert. At first he declined, and ultimately consented, only on the understanding that what he wrote should be revised by those who could have no personal bias in the matter, his one fear being—as he told the present writer in the last conversation which he had with him—that his own feelings might unconsciously lead him to do insufficient justice to his subject. That the performance of this kindly office must have gone far to soften the recollection of past animosities we may feel assured, and before the end came there was but little of bitterness left in the mind of the survivor. All three great workers now lie at rest in the same quiet country churchyard, their united work in the cause of scientific agriculture forming the most fitting and enduring monument of their labours, for its importance becomes every day more and more evident with the development of the superstructure which is being raised upon it.

Although Warington's original work in agricultural chemistry was brought to a close on his severance from Rothamsted, much useful work remained for him to do. The Committee of Management appointed him American lecturer under the Lawes Trust, and he consequently proceeded to the United States to perform his functions. The six lectures which he there delivered dealt chiefly

with the subject of nitrification, illustrated by his own work at Rothamsted. They were published by the United States Department of Agriculture.

On his return to England, Sir John Lawes invited him to carry out an investigation at his tartaric and citric acid factory at Millwall, on the contamination of these acids by the lead of the vessels used in their preparation. This Warington undertook, and he succeeded in finding a method for obviating the evil. He obtained, in addition, an excellent method for the accurate volumetric determination of lead in the acid. This formed the subject of a communication to the Society of Chemical Industry in 1893, the last communication of any investigation made by him.

In 1894 he was appointed one of the examiners in Agriculture under the Science and Art Department, and in the summer of the same year he was elected Sibthorpe Professor of Rural Economy at Oxford for three years.

The papers, other than those on original investigations, which Warington wrote, are numerous, and are all characterised by a lucidity of expression and precision of argument which renders them specially valuable. One of the most useful of his writings is, undoubtedly, a little volume entitled "The Chemistry of the Farm." The amount of appreciation with which it has been received, and the good which it has done, may be measured by the fact that it is now in its fifteenth edition, and is accepted as the text-book on the subject throughout the world, and as a model of what a text-book of that sort should be.

Warington continued to reside in Harpenden until the end. His habits and tastes did not predispose him to take any active part in village management, but whenever he thought that his knowledge might be of service to the community, he did not hesitate to give what assistance he could.

Educational or charitable work, however, always enlisted his sympathies and engaged his active support; whilst his strong religious convictions, guided by his clear judgment and absolute sincerity, rendered his church and philanthropic work peculiarly valuable. He certainly had an unusually high sense of public duty, and persistently throughout life did what he could to make his fellow-creatures better and happier. Missionary work always held a prominent place in his heart, as also did the training of the young, whether in religious or secular subjects, and during the last few years of his life much of his time and care was devoted to the Church day-schools. He was greatly interested in all work amongst the poor and needy, and was a liberal supporter of any organised charity which appealed to his judgment. Partly owing

to his isolated boyhood and youth, and partly to his lack of robust health, life went harder with him than it otherwise would have done, for the characteristics thus developed stood in his way, and often prevented his gaining the sympathy and appreciation which he was so ready to give to others.

Warrington was elected to the Chemical Society in 1863, and to the Royal Society in 1886. He served for two periods on the Council of the Chemical Society, and for one period as vice-president. For many years he was on the Library Committee of this Society, and did much useful work for the Fellows during the reorganisation and cataloguing of the books. For this his extensive acquaintance with chemical literature rendered him specially fitted.

Warrington was married twice. His first wife was a daughter of G. H. Makins, M.R.C.S., formerly chief Assayer to the Bank, and one of the Court of Assistants at the Society of Apothecaries. His second wife was a daughter of Dr. F. R. Spackman, who had for many years been medical practitioner at Harpenden. He has left five daughters by his first wife. In 1906 his health gave way, and he had a serious illness which necessitated a very difficult and dangerous operation. For this he prepared with singular equanimity and courage. The operation was successful; but though he nominally recovered from it, he never regained his strength, and eleven months afterwards (March 20th, 1907) he passed away.

SPENCER U. PICKERING.

AUGUST DUPRÉ.

BORN SEPT. 6TH, 1835; DIED JULY 15TH, 1907.

AUGUST DUPRÉ was born at Mainz on September 6th, 1835, and died at his residence, Mount Edgcumbe, Sutton, Surrey, after some weeks' illness, on July 15th, 1907, in his seventy-second year. He was the second son of J. F. Dupré, a merchant and citizen of the then Freie Reichsstadt of Frankfurt-am-Main, and his birth was entered in the register of the "Freie Französische Gemeinde" of that city. On his father's side Dupré traces his descent in a direct line from Cornelius Dupré, a French Huguenot who left France in 1685, after the suspension of the Edict of Nantes, and settled in the Palatinate, and who distinguished himself later as an officer in the army of Prince Eugene. Dupré's mother was also of Huguenot descent. His family was, therefore, originally French,

but by intermarriage had become practically German in the course of a hundred and fifty years.

Dupré had a somewhat varied school education, which he completed at the Polytechnic schools of Giessen and Darmstadt, and entered as a student of the University of Giessen in 1852, at the age of seventeen. There he studied chemistry under Professor Will, also attending the lectures of Kopp and others. From Giessen he proceeded to Heidelberg in 1854, Bunsen and Kirchhoff being among his teachers, and there he finally took his degree of Doctor of Philosophy in 1855, being barely twenty years old. It is interesting to note that fifty years later, in 1905, the University renewed his Diploma (Goldenes Doctor-Jubiläum) in recognition of his scientific work. Among his fellow students at Giessen and Heidelberg who became famous in later life were Harley, Matthiessen, Roscoe, and Volhard.

In the autumn of 1855 Dupré proceeded to London and became assistant to Odling, whom he accompanied to Guy's Hospital, remaining with him until 1863.

In 1864 he was appointed Lecturer on Chemistry and Toxicology at the Westminster Hospital Medical School, in succession to his elder brother, Dr. F. W. Dupré, who had given up the appointment in order to take up mining in the then recently discovered salt deposits at Stassfurt, in connexion with which he is now so well known.

August Dupré remained in London for the rest of his life, and became a naturalised English subject in 1866. He resigned his appointment at the Westminster Medical School in 1897, after thirty-three years' tenure, but during the last ten years, owing to pressure of consulting work, he had practically handed over the lectureship to the writer, who was associated with him as Assistant-Lecturer from 1885. From 1897 until his death in 1907 he continued to practise as consulting chemist, both privately and in connexion with several Government Departments, at his private laboratory in Edinburgh Mansions, Westminster.

Soon after he left the University Dupré began to publish various scientific papers, and, owing doubtless to this fact and the reputation for ability which he enjoyed in his own immediate circle, it was not long before he obtained several other public appointments in addition to the lectureship at Westminster.

Thus in 1871 he was appointed Chemical Referee to the Local Government Board, and about this time he was first consulted by Sir Vivian Majendie, then Colonel Majendie, Chief Inspector in the Explosives Department of the Home Office, to which Department he shortly after became permanently attached as Consulting

Chemist. In 1873 he became Public Analyst for Westminster, which post he held until 1901. In 1874 he was appointed Lecturer on Toxicology at the London School of Medicine for Women, an appointment in which he always showed the keenest interest and which he held until 1901.

He was also consulted by the Board of Trade, the Treasury, and the late Metropolitan Board of Works.

In all these appointments and consultations he may be said to have distinguished himself brilliantly by his rapid and thorough grasp of the problems in hand, his marked originality, his extreme conscientiousness, his intense enthusiasm, and his infinite capacity for taking trouble. He rapidly rose to eminence.

In 1875 he was elected a Fellow of the Royal Society. In 1877 he became President of the Society of Public Analysts. From 1871 to 1874 he sat on the Council of the Chemical Society. In 1885 he was made a Vice-President of the Institute of Chemistry. In 1886 he was elected Examiner in Chemistry to the Royal College of Physicians, and again in 1892.

In 1888 he was appointed a Member of the War Office Committee on Explosives, in 1891 an Associate Member of the Ordnance Committee, and in 1906 a Member of the Ordnance Research Board.

His earlier work for the Local Government Board, beginning in 1871, was largely analytical, but in 1884, 1885, and 1887 he made a series of investigations in connexion with the purification of water supplies by aëration and by the agency of bacteria, which must certainly rank as original researches of high merit and which undoubtedly have assisted greatly in the evolution of the most modern methods of treating sewage. They are published in the Medical Officers' Reports of the above dates, but are probably not widely known in the present day.

In conjunction with Abel, Dibdin, Keates, Odling, and Voelcker he advised the late Metropolitan Board of Works as to the condition of the Thames in 1878, 1882, and 1883, and in 1884 made numerous experiments in conjunction with Mr. Dibdin on the treatment of London sewage on a large scale. This work is referred to at great length in the Report of the Royal Commission on Metropolitan Sewage Discharge in 1884. He was a Member of the Departmental Committee on White Lead in 1893, and gave evidence before numerous other Royal Commissions.

Of all this Government work, it was the Home Office appointment which mainly occupied him. When, in 1871, he was first consulted by the Explosives Department, the manufacture in England of dynamite and guncotton had but recently commenced,

and these two were practically the only high explosives known at that time. Much had to be done on the part of the Government in connexion with the safe manufacture, storage, transport, and use of these explosives, and the rapid development of the industry necessitated the introduction of the Explosives Act of 1875. In 1876 the authorised list of explosives comprised twelve kinds only, but in 1907 it had risen to 182. In addition, during this period, 108 explosives had been passed by the Home Office after examination by Dupré, and over one hundred had been rejected by his advice. He thus investigated, during a period of thirty-six years, nearly four hundred entirely new explosives of the most varied composition, and further examined, at frequent intervals, all explosives imported into England as to safety. In the course of this work he had often to evolve original methods of analysis or of testing for safety, and in this latter direction especially he rendered great services to the Government and, indirectly, to the public.

It was also part of his duty to assist H.M. Inspectors in investigating the causes of various accidental explosions in factories and elsewhere, which occurred from time to time. His work, therefore, involved heavy responsibilities, and sometimes serious personal risks, notably during the Fenian outrages in 1882-83, when he had to examine several "infernal machines," and on the occasion of the Birmingham scare in 1883, when he superintended and himself assisted in the conversion of several hundred pounds of impure nitro-glycerine (which had been secretly manufactured in the heart of Birmingham) into dynamite, and so averted what might have been a terribly disastrous explosion. He was highly commended in the House of Commons by Sir William Harcourt, then Home Secretary, in connection with this "prompt and courageous action," and by Sir Vivian Majendie in the 8th Annual Report of the Inspectors of Explosives in 1883. As late as 1907 he devised a new method of testing for infinitesimal traces of mercury in explosive compounds. His private consulting work was also considerable, and he was engaged in many important law cases as a scientific witness.

It might well be supposed that these responsible undertakings engrossed him entirely, but this was far from being the case. During the first twenty years of his appointment at the Westminster Hospital Medical School he gave great attention to his lectures and to the practical teaching of chemistry. His lectures were always very fully illustrated with experiments, which year after year seemed to give him renewed pleasure to perform, and although not very easy to follow, he was always extremely interesting owing

to the mass of information he had ever ready to hand. In 1886 he published, in conjunction with the writer, then recently appointed Assistant-Lecturer, "A Manual of Inorganic Chemistry," which had some success, and which reached its third edition in 1901. This book was dedicated to Professor Will, of Giessen, whom he always spoke of with the highest admiration and reverence as a great teacher.

The subject of toxicology, on which, as already said, he also lectured both at Westminster and at the London School of Medicine for Women, had always specially interested him, and he became known and was not unfrequently consulted as a toxicologist. He was brought into particular prominence in connexion with the celebrated Lamson case in 1881.

As an instance of the thoroughness of his work, the writer well remembers Dupré tasting sixteen quinine powders which had been prepared for the unfortunate victim in this case, and his almost immediately experiencing the now familiar and somewhat alarming physiological effect of the aconitine which he found in the last powder. He was associated in this case with Sir Thomas Stevenson.

It has already been mentioned that very soon after leaving the University Dupré began to publish scientific papers, and it seems surprising that amid such varied occupations he found time to work out so many original problems. His papers amount to no less than thirty-four in number between 1855 and 1902. Of these, five papers are included in the Proceedings and Transactions of the Royal Society between 1866 and 1872. The first, in 1866, with Dr. Bence Jones, on "Animal Quinoidine," may be said to have anticipated the later important researches of Selmi and others on Ptomaines. Another, in 1871, dealt ably with the Elimination of Alcohol in the human subject, a problem then arousing much interest. The remaining four papers, published between 1868 and 1872, some of the work being done in conjunction with the late Mr. F. J. M. Page, rank, perhaps, as his best efforts, treating of the specific heat and other characters of various aqueous mixtures and solutions, notably of mixtures of ethyl alcohol and water, in the course of which he made the remarkable observation that mixtures of these last two substances up to 36 per cent. of ethyl alcohol had a specific heat sensibly higher than that of water itself.

In the Journal of the Chemical Society are found eight papers between 1867 and 1880. One on the Synthesis of Formic and Sulphurous Acids, four on the Various Constituents of Wine, including compound ethers, one on the Estimation of Urea with Hypobromite by means of an ingenious apparatus now so universally em-

ployed, and two, in conjunction with the writer, on a New Method of Estimating Minute Quantities of Carbon, which was included by the late Dr. E. Frankland in his well-known work on Water Analysis.

Between 1877 and 1883 he read no less than thirteen papers before the Society of Public Analysts dealing with the analysis of foods or water, and most of the methods evolved by him in these publications are still used or have given rise to improved operations, notably those dealing with butter fat, fusel oil in whiskey and other spirits, alum in flour and bread, foreign colouring matters in wine, and methods of water analysis.

He published only two papers on Explosives, to which he had given such great attention, before the Society of Chemical Industry, and these as late as 1902. As a matter of fact, however, much original work was done by him in this branch of chemistry, some of which appears in the Annual Reports of H.M. Inspectors of Explosives, while again much could not be put forward owing to his official connexion with the Home Office.

His earliest papers, published between 1855 and 1862, are six in number, and deal with volumetric methods and spectrum analysis (conjointly with his brother, Dr. F. W. Dupré), the iodic test for morphia, and the presence of copper in plant and animal tissues, this last in conjunction with Odling.

To the chemistry of wine, as will be seen from the above summary, he devoted a good deal of attention, and was joint author with Dr. Thudichum of a work entitled "On the Origin, Nature, and Varieties of Wine," published in 1872, in which a considerable amount of original analytical work is embodied.

Dupré married, in 1876, Miss Florence Marie Robberds, of Manchester, and leaves a family of one daughter and four sons, two of whom, Frederick and Percy, are now carrying on his work for the Home Office. He was of a striking personality, of medium height, but very powerfully built, with a massive head and brow, and must have possessed an iron constitution. As a young man he was a skilled fencer and swimmer. He was of somewhat excitable temperament, but had a most kindly disposition. Although not a fluent speaker, he was impressive from his obvious sincerity, and the thorough knowledge he displayed. He therefore made an excellent expert witness, and was more than once complimented in Court on his straightforward evidence. In controversy he was unsparing where facts were concerned, and at times intensely sarcastic.

Although almost wholly devoted to chemistry, his mind found many other outlets. He was a great student of history, and his quite remarkable memory was frequently exemplified in conversa-

tion on this subject. He was also exceptionally well read in general as well as in scientific literature, both English and German, and amassed a large collection of books. Among other hobbies he pursued astronomy and photography. His mind, indeed, seems rarely to have been idle; he had a perfect passion for work, and, except for a few weeks' holiday annually, he never relaxed. There is little doubt that at one time, about 1891, he overstrained his brain, and was obliged for some months to take a complete rest, which, fortunately, restored him to renewed energy. Like many great men, he was of a modest and retiring nature, and probably but few of his contemporaries have realised the magnitude and variety of the work he accomplished during fifty years of almost unceasing activity.

H. WILSON HAKE.

JOHN CLARK, PH.D., F.I.C.

BORN 1844; DIED JULY 9TH, 1907.

DR. CLARK was born in 1844, being the only son of John Clark, a solicitor of eminence in the City of Glasgow. He received his education in the classics at Glasgow University, and during his period of study there acquired a taste for chemistry and became a pupil in the laboratory of the late Dr. Frederick Penny, who was successor to Graham, Ure, and Gregory in the Chair of Chemistry of Anderson's College, now incorporated in the Glasgow and West of Scotland Technical College. He subsequently proceeded to the University of Göttingen, where he worked with Fittig and Wöhler, gaining the degree of Doctor of Philosophy for a dissertation on amidovalerianic acid. He also studied for a session at Heidelberg under Bunsen, and afterwards worked in Paris for nine months in the laboratory of Prof. Payen at the Conservatoire des Arts et Metiers. At the still early age of twenty-three he returned to Glasgow, where he was for three years senior assistant at the Andersonian College to his old teacher, Penny, acting as his substitute during the illness which ended in Penny's death. In 1870 he joined his friends, Mr. Tatlock and the late Dr. Wallace, in forming the widely-known firm of Wallace, Tatlock and Clark, who, in addition to their analytical practice, carried on a very successful private school of technical chemistry. For some years Dr. Clark also lectured on chemistry in the Medical School of the Royal Infirmary at Glasgow. In 1888 the original partnership was dissolved by the retirement from the firm of Mr. Tatlock, who estab-

lished the separate practice which he still carries on in conjunction with Mr. R. T. Thomson; and the death of Dr. Wallace left Dr. Clark in sole charge of the laboratory of the original firm at 138, Bath Street, until his son and survivor, Mr. R. M. Clark, became qualified, a few years since, to join his father in partnership.

Dr. Clark's contributions to chemical literature were many, being almost wholly directed to the practical advancement of analytical chemistry. In the *Analyst* only one paper appears to have been published, namely, one on the "Composition of Dutch Butter," 1901. In the *Journal of the Chemical Society* he published the following papers:—"Estimation of Phosphoric Acid with Nitrate of Silver," 1888; "Separation of Arsenic, Antimony, and Tin," 1892; "The Use of Sodium Peroxide as an Analytical Reagent," 1893; "Fleitman's Test with Arsenic Acid," 1893; "Improvements in Reinsch's Test for Arsenic," 1893.

In the *Journal of the Society of Chemical Industry*:—"Composition of Tobacco," 1884; "New Method of Estimating Sulphur in Pyrites," 1885; "New Method of Estimating Arsenic in Pyrites," 1887; "Alloys of Aluminium," 1887 and 1891; "Transvaalite, a New Cobalt Mineral," 1890; "Analysis of Copper, &c.," 1900; "Separation of Bismuth from Lead," 1900; "Direct Estimation of Arsenic in Minerals, Metals, &c.," 1891; "Estimation of Chromium in Steel," 1892; "Estimation of Chromium in Ferro-Chromium and Steel," 1892; "Determination of Arsenic in Alkaline Solution," 1893; "Estimation of Nickel and Zinc as Phosphate," 1896; "Estimation of Antimony in Ores and Metals," 1896.

In the *Journal of the Philosophical Society of Glasgow*:—"Action of Phosphuretted Hydrogen on the Animal Organisms," 1879; "Volumetric Process for the Estimation of Cobalt and Nickel," 1883; "A New Process for the Estimation of Nickel and Cobalt," 1883.

In the *Chemical News*:—"Estimation of Chromium," 1871.

Among the public appointments he held were the Public Analystships for the counties of Lanark and Renfrew, and the burghs of Ayr, Kilmarnock, Girvan, Dumbarton, Kinning Park, Motherwell, Partick, Barrhead, Paisley, Renfrew, and Dornoch, and for the City of Glasgow, the last-named appointment being held conjointly with Mr. Tatlock and Mr. Harris.

At the time of his death Dr. Clark was President of the Association of Public Analysts of Scotland, as well as of the parent Society of Public Analysts, and a member of the Council of the Institute of Chemistry, and he had filled the office of Chairman of the Scottish Section of the Society of Chemical Industry.

His acquisition of French and German at an early age enabled him to read, write, and speak these languages with facility, and to keep himself abreast of the chemical literature of the Continent.

Although fully occupied in his professional life, he found time and opportunity for physical recreation of various kinds, golfing, bowling, and angling, in all of which he excelled, and in this capacity received presidential honours from the clubs and associations with which he was connected. During his German University student days he was a sufficiently orthodox student to earn the reputation of a keen duellist, and in moments of early reminiscence he was still proud of the scars which constituted the lasting trophies of this mimic but sanguinary warfare. His adventures in this direction must be put down to his love of sport rather than to any natural tendency to quarrel, for his disposition was one of the kindest and most genial, and his bright face and physically handsome presence will be long missed in the circles in which he personally moved. Few, probably, have gained greater respect than he commanded, both within his profession and in the eye of the public, and the loss of his friendship, as well as of his ever-ready advice and assistance, will be widely felt.

R. R. TATLOCK.

FREDERICK JAMES MONTAGUE PAGE.

BORN JUNE 27TH, 1848; DIED AUG. 16TH, 1907.

FREDERICK JAMES MONTAGUE PAGE was born at Chelmsford on June 27th, 1848, being an only child. When he was eight years old he came to London with his parents, and in due course entered the City of London School, at that time in Milk Street. While there, he carried off many prizes and medals, and obtained the "John Carpenter" Scholarship. In 1866, when eighteen years of age, he gained an exhibition to the Royal School of Mines, where he studied under Huxley, Tyndall, Frankland, and Percy. The following year he was first in chemistry and in physics, and at the close of his three years' training, 1869, he took the associateship of the Royal School of Mines, again passing first in chemistry and first in physics. He took his final B.Sc. London in the same year, having passed the preliminary (first class) in 1868 with honours in chemistry and "natural philosophy."

His first appointment on leaving the Royal School of Mines was that of assistant gas examiner to the Corporation of the City of London, but in 1870 he went to Dr. Thudichum as his assistant, where he was occupied for about three years in chemical research

undertaken for the Medical Department of the Privy Council. In 1873 he left Dr. Thudichum to become the assistant of Dr. Burdon Sanderson, first at the Brown Institute and subsequently at University College, remaining with him until the year 1883, when he was appointed lecturer in physics and demonstrator in practical chemistry to the London Hospital—appointments he held at the time of his death. During the winter of 1879 and 1880 he delivered courses of lectures on physics and chemistry at the Royal Gardens, Kew, and from 1880 to 1906 he gave lectures on chemistry and physics at the establishment of the well-known tutors, Messrs. Wren and Gurney. He was for two years assistant examiner in physiology at London University, and also held an examinership at the Society of Apothecaries.

Page was with the writer of this obituary at the Birmingham meeting of the Society of Chemical Industry in July, 1907, and then seemed to be in bad health, but put aside the suggestion that he should consult a medical man. In August he went to Weymouth for a holiday, and, being an excellent swimmer and fond of the sport, he went into the sea, but became unconscious and was brought ashore. He was attended by three resident physicians, and his colleague, Dr. Head, of the London Hospital, also came down to see him, but he never recovered consciousness, and died of cerebral hæmorrhage on August 16th, 1907, ten days after the attack.

His contributions to science were more physiological than chemical; amongst them are a paper "On the Specific Heats of Mixtures of Ethyl Alcohol and Water," published in the *Phil. Trans.*, 1869, p. 591, in collaboration with Dr. A. Dupré; one "On the Influence of Surrounding Temperature on the Discharge of Carbonic Acid in the Dog"; and four papers in conjunction with Sir Burdon Sanderson, one being "On Mechanical Effects and on the Electrical Disturbance Consequent on Excitation of the Leaf of *Dionea muscipula*," *Proc. Roy. Soc.*, 1877, **25**, 4, and the others published in *Proc. Roy. Soc.*, 1877, **25**, 411; 1878, **27**, 410; 1880, **30**, 373; and in *J. Physiol.*, **2**, p. 384, "On Excitatory Processes on the Ventricle of the Heart of the Frog." His only contribution to our Journal was in 1876, i, p. 24, describing a simple gas regulator for thermostats. In conjunction with Dr. Luff he produced a "Manual of Chemistry" and also a text-book on "Elementary Physics."

He served on the Council of the Chemical Society, and five times on the Council of the Institute of Chemistry. He was also a member of the Society of Chemical Industry and of the Physiological Society.

He was an enthusiastic musician, no mean performer on the piano, and in his younger days had a fine tenor voice—this was sweet and sympathetic even up to the time of his decease.

He was a staunch Churchman, and member formerly of the choir of St. Martin's-in-the-Fields, and subsequently of St. Peter's, Eaton Square. He loved "part singing," and was for many years a member of the well-known "Moray Minstrels" and also of the "City Glee Club," of which he had been elected president shortly before his death. He was a member of the John Carpenter Club, holding the office of president in 1902.

As a man, all those who knew him well held him in high esteem, he was ever ready to do a kindness, and that not merely to his intimate friends; his genial manner, ready wit, and sterling good sense will long live in the memory of many of us.

C. E. G.

SIR DAVID GAMBLE, BART., K.C.B.

BORN FEB. 3RD, 1823; DIED FEB. 4TH, 1907.

SIR DAVID GAMBLE, Bart., K.C.B., was born on February 3rd, 1823, in Dublin. His father, Josias Christopher Gamble, was descended from an Ayrshire family, which removed to Lisbellaw, near Enniskillen, in 1620. Jos. C. Gamble removed with his family to Lancashire in 1828 to find a suitable site for chemical works. This he found at St. Helens, on the banks of the St. Helens Canal.

David Gamble went to school at Cowley Hill, St. Helens, kept by a Mr. Morley, and afterwards at Runcorn. On leaving school he studied chemistry at University College, London, under Thomas Graham, and afterwards at the Andersonian Institution, Glasgow. While in Glasgow he made the acquaintance of the Tennant family, with whom the firm had business relations. In 1842, at the age of nineteen, he joined his father's firm, which then became Jos. C. Gamble & Son.

Mr. Gamble was preparing bleach at the Gerards Bridge Works, but the anxieties connected with the disputed validity of one of his patents, added to the claims continually made by landowners and agriculturists on account of damage done by escaping hydrochloric acid, had so told on his health that David on coming into the firm almost immediately assumed charge. In 1846 the firm was Gamble, Son and Sinclair, and very soon afterwards became again Jos. C. Gamble & Son. At this time they manufactured alum, as well as the products usually made at alkali works. The firm was one of the first and largest to manufacture Epsom salts on a large

scale from carbonate of magnesia imported from Greece. This was, about the 'sixties, much used for weighting calico.

David Gamble married Elizabeth Haddock, in 1847, and resided near to the works, ultimately building the mansion "Windlehurst" in 1860. His eldest son, Josias Christopher Gamble, the second baronet, who died soon after succeeding to the title, joined the firm in 1867, which was at that time the first to carry out Weldon's process for the recovery of manganese on a large industrial scale. The firm was now Jos. C. Gamble & Son. They were also one of the first to make potassium chlorate on a large scale, and about 1869 they bought the Hardshaw Brook Works, where they manufactured chiefly saltcake, bleach, and potassium chlorate. They also manufactured for a short time chlorates of barium, aluminium, etc. The firm carried on operations on a very large and important scale with increasing success, and when the United Alkali Co., Ltd., was formed, in 1890, there was some difficulty in inducing Messrs. Jos. C. Gamble and Son to join. However, in 1891 they joined the United Alkali Co., which thus secured a practical monopoly in Great Britain of alkali manufacture and kindred industries.

Sir David Gamble was one of the most active members of the committee which raised funds for the establishment of the Volunteer force in St. Helens in 1859 and 1860. He was captain of the first company, and as the force grew he was promoted to be major, in which capacity he served so earnestly and with so much skill that this force became one of the best equipped and trained units in the country. Ultimately he became lieutenant-colonel. It was his generosity which provided a drill-hall and parade-ground; in short, he provided in every way for the efficiency of the 47th Lancashire Volunteers during the twenty-seven years that he was its commanding officer. He retired in 1887, becoming honorary colonel. He was also very fond of the sea, which he enjoyed in his own yacht. He was a leading member of the Royal Mersey Yacht Club for forty-nine years, becoming vice-commodore in 1873 and commodore in 1882, a position which he retained until his death in 1907. Residing as he did near St. Helens, in the midst of a community almost entirely engaged in manufactures, he paid a great deal of attention to organising and improving the condition of the town and its inhabitants. Taking a leading part in obtaining the Improvement Act, 1845, he became Chairman of the Improvement Commissioners, and when in 1868 St. Helens was incorporated, Colonel Gamble became the first mayor, a position to which he was re-elected twice in successive years. He was also mayor in 1882-3, and again in 1886-7.

On the occasion of Queen Victoria's Jubilee in 1887 he was made Commander of the Bath. He was created a baronet in 1897 and K.C.B. in 1904.

Sir David Gamble took a leading part in the foundation of the University College, Liverpool, which afterwards became the University of Liverpool. He not only contributed liberally and repeatedly to its funds, but devoted time and attention to its interests as a member of the Court of Governors as well as privately. His interests were not confined to the Chemical Department, although to it he was on many occasions a good friend. He was always willing in the most courteous way to listen to appeals, whether for help or advice, and many important advances were due in great measure to his wisdom, his sympathy, and his generosity.

In 1868 Sir D. Gamble built the Windle Schools at Cowley Hill, St. Helens. He was a governor of Cowley Schools, and promoted the extension of these schools, and also built and equipped a high-class technical school and free library for St. Helens, known as the Gamble Institute.

During sixty-four years of active industrial and public life, Sir David Gamble was characterised by the great consideration and courtesy which he extended to everyone with whom he had to do either in a public or private capacity. Possessed of great ability, he spent his energies more for others than for himself. His thoughtful care for the workpeople around him led him into schemes for their benefit far too many to be enumerated. His work and gifts were bestowed in the most unostentatious manner. Besides his activity in the public service as a magistrate and otherwise, his business ability made him a valued director of Parr's Bank from its foundation, and of other companies. He was a partner in iron works at Ditton. He and Mr. Henry Deacon built and started the works at Widnes which became the Tharsis Sulphur and Copper works there.

And when, on February 4th, 1907, the day after his eighty-fourth birthday, he passed away full of years and still active, the whole community of St. Helens, the County of Lancashire, and innumerable friends far beyond the boundaries of the county felt that they had suffered an irreparable loss.

J. CAMPBELL BROWN.

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$C_2H_2N_6$	1071	$C_5H_3O_2NCl_2 \cdot 3H_2O$	2000	$C_7H_7O_2Br$	791, 792
C_2N_4Ta	2176			$C_7H_7O_2N$	570
$C_2H_4ON_3$	1870	C₆ Group.		$C_7H_{10}O_2Br_2$	588, 590
$C_2H_4O_2N_3$	77	$C_6H_6O_4$	721	$C_7H_{10}O_2Ba$	1782
$C_2H_4ON_4$	80	$C_6H_6O_4$	598, 600	$C_7H_{11}O_2Br$	585
$C_2H_4ON_5$	1867	$C_6H_{10}O_{25}H_2O$	589	$C_7H_{11}O_2N$	583
$C_2H_4O_2N_3Ag$	78	$C_6H_{10}O_6$	720, 722, 723	$C_7H_{13}O_2N_3H_2O$	539
		$C_6H_{16}N_6$	1178	$C_7H_{13}O_2Ag$	1739
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C_3H_2N	1178, 1179	$C_6H_4O_2N_4$	1382	$C_7H_6O_4NBr$	792
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$C_3H_2O_2N_3$	81, 671, 1861	$C_6H_7O_4Ag$	601	$C_7H_2O_4AsNa \cdot 2H_2O$	1896
$C_3H_2N_2Br_2$	1178	$C_6H_7O_4As$	1895	$C_7H_7O_2N_2S$	626
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$C_3N_2S_3B$	2178	$C_6H_7O_4Na$	601	1405	
$C_3H_4O_2N_3Ag$	672	$C_6H_8O_6Ag_2$	720, 721, 723, 725	$C_7H_6O_2NS_2Na \cdot H_2O$	1404
$C_3ON_2S_3P$	2157			$C_7H_3O_3NAsNa \cdot 5H_2O$	1181
C₄ Group.		$C_6H_{12}O_4N_2$	720, 724		
$C_4H_4O_2$	946, 947	$C_6H_2O_7N_3Cs$	477	C₈ Group.	
$C_4H_4O_2N_6$	1074	$C_6H_2O_7N_3Li \cdot H_2O$	475	C_8H_{12}	644
$C_4H_6O_3S$	1834	$C_6H_2O_7N_3Li \cdot 4H_2O$	474, 475	$C_8H_4O_2$	569
$C_4H_4ON_3$	675, 676, 677	$C_6H_2O_7N_3Na \cdot H_2O$	476	$C_8H_{10}O_9$	1781
$C_4H_4O_2N_3$	79, 1868	$C_6H_3O_2NCl_2$	1995	$C_8H_{11}N$	1959
$C_4H_4ON_4$	678	$C_6H_4ON_2Cl_2$	1996	$C_8H_{12}O$	1969
$C_4H_4ON_6$	83	$C_6H_4O_2N_2Br_2$	734	$C_8H_{10}O_2$	1422, 1423, 1884, 1886, 1960, 1961
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				$C_8H_{12}Br_4$	650
C₅ Group.		C₇ Group.		$C_8H_{14}O$	641
$C_5H_2N_2Cl$	1380	$C_7H_6O_4$	1027, 1028	$C_8H_{14}O_3$	1882, 1884
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$C_5H_4ON_6$	676, 678	$C_7H_{12}O_3$	584	$C_8H_{12}Br$	1078
$C_5H_{10}O_2N_6$	83	$C_7H_{12}O_5$	1783	$C_8H_{16}O$	1078
		$C_7H_{14}O_3$	1789		

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1563	$\text{H}_{12}\text{O}_2\text{NNa}$
85	$\text{H}_{12}\text{O}_2\text{N}_2\text{S}$
260	$\text{H}_{12}\text{O}_2\text{ClBr}$
868	H_{12}ONCl
1888	H_{12}ONCl
527	$\text{H}_2\text{OCl}_2\text{SHg}$
21	$\text{H}_{11}\text{O}_2\text{N}_2\text{BrS}$

C₁₁ Group.

2018, 2019, 2020	H_{11}O_2
182, 1515	H_{12}O_2
1028	H_{12}O_2
867	H_{12}O_2
1084, 1968	H_{12}O_2
1968	H_{12}O_2
1382	$\text{H}_2\text{O}_3\text{N}_4$
1380	$\text{H}_2\text{N}_2\text{Cl}$
180, 182	H_2ON
1378, 1379	H_2ON_3
529	H_2OS
529	$\text{H}_{11}\text{O}_2\text{N}$
1956, 1958, 1962, 1973	$\text{H}_{12}\text{O}_2\text{N}$
1806	$\text{H}_{12}\text{O}_2\text{N}$
1295	$\text{H}_{12}\text{OBr}_2$
1297, 1298	H_{12}OBr
1295	$\text{H}_{12}\text{O}_2\text{S}$
1298	H_{12}ON
1082	$\text{H}_{12}\text{O}_2\text{Br}$
38	$\text{H}_{12}\text{O}_2\text{N}_3$
37	$\text{H}_{12}\text{O}_2\text{N}_3$
101	$\text{H}_2\text{O}_2\text{N}$
528	$\text{H}_{12}\text{O}_2\text{Cl}_2\text{Hg}$
678	$\text{H}_{11}\text{O}_2\text{N}_2\text{S}$
1297	$\text{H}_{12}\text{O}_2\text{NS}$
1806	$\text{H}_{12}\text{O}_2\text{NCl}$
1296	$\text{H}_{12}\text{O}_2\text{ClS}$
626	$\text{H}_{12}\text{O}_2\text{NS}$
1297	$\text{H}_{12}\text{O}_2\text{NS}$
1299	H_2ONCl
529	$\text{H}_{12}\text{OCl}_2\text{SHg}$
1807	$\text{H}_{12}\text{O}_2\text{NCl}_4\text{Au}$

C₁₂ Group.

1189, 1190	H_2O_7
1191	$\text{H}_2\text{O}_7\cdot 2\text{H}_2\text{O}$
434	H_{12}O_2
2019, 2020	H_{12}O_2

1283	$\text{C}_{12}\text{H}_{12}\text{O}_3$
1158	$\text{C}_{12}\text{H}_{12}\text{O}_4$
1025, 1026	$\text{C}_{12}\text{H}_{14}\text{O}_8$
724	$\text{C}_{12}\text{H}_{14}\text{O}_9$
579, 582	$\text{C}_{12}\text{H}_{18}\text{O}_3$
1787	$\text{C}_{12}\text{H}_{22}\text{O}_6$
478	$\text{C}_{12}\text{H}_4\text{O}_{14}\text{Mg}, 9\text{H}_2\text{O}$
609	$\text{C}_{12}\text{H}_6\text{O}_6\text{N}_6$
610, 611	$\text{C}_{12}\text{H}_7\text{O}_4\text{N}_2$
1188	$\text{C}_{12}\text{H}_7\text{O}_2\text{Na}$
1185	$\text{C}_{12}\text{H}_7\text{O}_2\text{Na}, 4\text{H}_2\text{O}$
1185	$\text{C}_{12}\text{H}_7\text{O}_2\text{K}$
1188	$\text{C}_{12}\text{H}_7\text{O}_2\text{K}, 3\text{H}_2\text{O}$
611, 612	$\text{C}_{12}\text{H}_8\text{O}_2\text{N}_4$
1019	$\text{C}_{12}\text{H}_9\text{O}_2\text{N}_3$
2100	$\text{C}_{12}\text{H}_9\text{O}_2\text{N}_6$
2100	$\text{C}_{12}\text{H}_9\text{O}_{16}\text{N}_2$
1384	$\text{C}_{12}\text{H}_{10}\text{O}_2\text{N}_2$
1526	$\text{C}_{12}\text{H}_{10}\text{O}_2\text{S}_2$
177, 181, 183, 1284	$\text{C}_{12}\text{H}_{11}\text{ON}$
1153	$\text{C}_{12}\text{H}_{11}\text{O}_3\text{Cl}$
530	$\text{C}_{12}\text{H}_{12}\text{OS}$
185, 530	$\text{C}_{12}\text{H}_{13}\text{ON}$
1964, 1965, 1973	$\text{C}_{12}\text{H}_{17}\text{O}_2\text{N}$
251	$\text{C}_{12}\text{H}_{19}\text{O}_2\text{N}$
1367	$\text{C}_{12}\text{H}_{27}\text{O}_3\text{As}$
326	$\text{C}_{12}\text{HONBr}_8$
325	$\text{C}_{12}\text{H}_2\text{ONBr}_7$
326	$\text{C}_{12}\text{H}_3\text{ONBr}_6$
323	$\text{C}_{12}\text{H}_4\text{ONBr}_5$
325	$\text{C}_{12}\text{H}_5\text{ONBr}_4$
479	$\text{C}_{12}\text{H}_4\text{O}_{14}\text{N}_6\text{Ca}$
479	$\text{C}_{12}\text{H}_4\text{O}_{14}\text{N}_6\text{Ca}, 10\text{H}_2\text{O}$
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$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2$	705
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2$	709
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{H}_2\text{O}$	704
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{H}_2\text{O}$	704
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}$	709
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{H}_2\text{O}$	704
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2$	1932
$\frac{1}{2}\text{H}_2\text{ON}$	1630
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{S}_2\text{H}_2\text{O}$	1625
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{S}_2\text{H}_2\text{O}$	1625
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{S}_2\text{H}_2\text{O}$	1625
$\frac{1}{2}\text{H}_2\text{O}_3\text{ClS}_2\text{H}_2\text{O}$	762
$\frac{1}{2}\text{H}_2\text{O}_3\text{N}_2\text{S}$	1624
$\frac{1}{2}\text{H}_2\text{O}_3\text{NBrS}$	1238

	PAGE
C₂₈ Group.	
$\text{C}_{28}\text{H}_{20}\text{O}_4\text{N}_2$	1941
$\text{C}_{28}\text{H}_{30}\text{OSi}_2$	208
$\text{C}_{28}\text{H}_{30}\text{O}_3\text{N}_2\text{H}_2\text{O}$	1393
$\text{C}_{28}\text{H}_{31}\text{O}_4\text{N}_2$	1392
$\text{C}_{28}\text{H}_{47}\text{ON}_2$	1629
$\text{C}_{28}\text{H}_{36}\text{O}_4\text{NBrS}$	1235
C₂₉ Group.	
$\text{C}_{29}\text{H}_{17}\text{N}$	1764
$\text{C}_{29}\text{H}_{20}\text{O}_2$	953
$\text{C}_{29}\text{H}_{46}\text{O}_4$	1683
$\text{C}_{29}\text{H}_{48}\text{O}_4$	1682, 1685
$\text{C}_{29}\text{H}_{46}\text{O}_2$	1631
$\text{C}_{29}\text{H}_{30}\text{O}_2$	1686
$\text{C}_{29}\text{H}_{30}\text{O}_6\text{N}_2\text{S}_2\text{H}_2\text{O}$	1623
$\text{C}_{29}\text{H}_{32}\text{O}_7\text{N}_2\text{S}_2\text{H}_2\text{O}$	1623
$\text{C}_{29}\text{H}_{36}\text{O}_3\text{N}_2\text{S}$	1625
C₃₀ Group.	
$\text{C}_{30}\text{H}_{38}\text{O}_4$	953
$\text{C}_{30}\text{H}_{38}\text{O}_2$	912
$\text{C}_{30}\text{H}_{30}\text{O}_6\text{N}_2\text{H}_2\text{O}$	1390
$\text{C}_{30}\text{H}_{30}\text{O}_2\text{N}_2$	1391
$\text{C}_{30}\text{H}_{33}\text{O}_6\text{N}_2\text{H}_2\text{O}$	1390
$\text{C}_{30}\text{H}_{10}\text{O}_6\text{N}_2\text{Cr}_2\text{H}_2\text{O}$	486
$\text{C}_{30}\text{H}_{10}\text{O}_3\text{N}_2\text{Cr}_2\text{H}_2\text{O}$	486
$\text{C}_{30}\text{H}_{20}\text{O}_2\text{Cl}_2\text{Pt}$	1110
$\text{C}_{30}\text{H}_{22}\text{O}_4\text{Cl}_2\text{Pt}$	1099
$\text{C}_{30}\text{H}_{23}\text{O}_4\text{Cl}_2\text{Pt}_2\text{H}_2\text{O}$	1099
$\text{C}_{30}\text{H}_{34}\text{O}_6\text{N}_2\text{S}$	1623
$\text{C}_{30}\text{H}_{34}\text{O}_7\text{N}_2\text{S}_2\text{H}_2\text{O}$	1624
C₃₁ Group.	
$\text{C}_{31}\text{H}_{48}\text{O}_2$	898
$\text{C}_{31}\text{H}_{50}\text{O}_2$	897
$\text{C}_{31}\text{H}_{50}\text{O}_2$	1681, 1931
$\text{C}_{31}\text{H}_{34}\text{O}_6\text{N}_2\text{H}_2\text{O}$	1390
$\text{C}_{31}\text{H}_{36}\text{O}_6\text{N}_2\text{S}_2\text{H}_2\text{O}$	1624
$\text{C}_{31}\text{H}_{36}\text{O}_7\text{N}_2\text{S}_2\text{H}_2\text{O}$	1624
$\text{C}_{31}\text{H}_{36}\text{O}_3\text{N}_2\text{S}_2\text{H}_2\text{O}$	1624
C₃₂ Group.	
$\text{C}_{32}\text{H}_{20}\text{O}_2$	899
$\text{C}_{32}\text{H}_{20}\text{O}_2$	912
$\text{C}_{32}\text{H}_{30}\text{O}_6\text{N}_2$	703
$\text{C}_{32}\text{H}_{34}\text{O}_6\text{N}_2$	1391

	PAGE
$\text{C}_{32}\text{H}_{36}\text{O}_7\text{N}_2\text{H}_2\text{O}$	1391
$\text{C}_{32}\text{H}_{38}\text{O}_6\text{N}_2\text{H}_2\text{O}$	702
$\text{C}_{32}\text{H}_{38}\text{OSi}_2$	450
$\text{C}_{32}\text{H}_{38}\text{O}_{13}\text{N}_2$	1805
$\text{C}_{32}\text{H}_{38}\text{O}_7\text{N}_2$	1391
$\text{C}_{32}\text{H}_{22}\text{O}_2\text{Cl}_2\text{Pt}$	1100
$\text{C}_{32}\text{H}_{22}\text{O}_4\text{Cl}_2\text{Au}$	1101
$\text{C}_{32}\text{H}_{22}\text{O}_4\text{Cl}_2\text{Pt}_2\text{H}_2\text{O}$	1101
$\text{C}_{32}\text{H}_{36}\text{O}_4\text{Cl}_2\text{Pt}$	1112
C₃₃ Group.	
$\text{C}_{33}\text{H}_{27}\text{O}_7$	1606
$\text{C}_{33}\text{H}_{22}\text{O}_4$	899
$\text{C}_{33}\text{H}_{54}\text{O}_5$	1682
$\text{C}_{33}\text{H}_{32}\text{O}_5\text{N}_2\text{H}_2\text{O}$	1393
$\text{C}_{33}\text{H}_{33}\text{O}_4\text{N}_2\text{H}_2\text{O}$	1392
$\text{C}_{33}\text{H}_{34}\text{O}_7\text{N}_2\text{H}_2\text{O}$	1392
$\text{C}_{33}\text{H}_{36}\text{O}_3\text{N}_2\text{H}_2\text{O}$	1392
$\text{C}_{33}\text{H}_{46}\text{ON}_2$	1932
$\text{C}_{33}\text{H}_{50}\text{O}_2\text{N}_2$	1634
C₃₄ Group.	
$\text{C}_{34}\text{H}_{30}\text{O}_7$	955, 956
$\text{C}_{34}\text{H}_{30}\text{O}_8$	1607
$\text{C}_{34}\text{H}_{52}\text{O}_2$	1632
$\text{C}_{34}\text{H}_{54}\text{O}_4$	899
$\text{C}_{34}\text{H}_{49}\text{O}_2\text{N}$	1930
$\text{C}_{34}\text{H}_{28}\text{O}_4\text{Cl}_2\text{Pt}$	434
$\text{C}_{34}\text{H}_{30}\text{O}_3\text{Cl}_2\text{Fe}_2\text{H}_2\text{O}$	1151
$\text{C}_{34}\text{H}_{30}\text{O}_8\text{Cl}_2\text{Pt}$	1114
$\text{C}_{34}\text{H}_{40}\text{O}_8\text{N}_2\text{Cl}_2\text{Pt}$	1807
C₃₅ Group.	
$\text{C}_{35}\text{H}_{64}\text{O}_2$	898
$\text{C}_{35}\text{H}_{68}\text{O}$	911, 913
$\text{C}_{35}\text{H}_{68}\text{O}_2$	906
$\text{C}_{35}\text{H}_{70}\text{O}_2$	910
$\text{C}_{35}\text{H}_{38}\text{O}_5\text{N}_2\text{H}_2\text{O}$	1392
C₃₆ Group.	
$\text{C}_{36}\text{H}_{30}\text{O}_6\text{Cl}_2\text{Pt}_2\text{H}_2\text{O}$	1104
$\text{C}_{36}\text{H}_{30}\text{O}_8\text{Cl}_2\text{Pt}_2\text{H}_2\text{O}$	1106, 1148
$\text{C}_{36}\text{H}_{24}\text{O}_{17}\text{N}_2\text{S}_2\text{Cr}_2$	1694
$\text{C}_{36}\text{H}_{24}\text{O}_{10}\text{N}_6\text{Cl}_2\text{Pt}_2$	1694
C₃₇ Group.	
$\text{C}_{37}\text{H}_{72}\text{O}_2$	906
$\text{C}_{37}\text{H}_{84}\text{O}_2\text{N}_2$	711

	PAGE		PAGE
$C_{37}H_{36}O_9N_2H_2O$	1391	C₄₂ Group.	
$C_{37}H_{37}O_7N_2, 4\frac{1}{2}H_2O$	1391	$C_{42}H_{28}O_{10}$	738
$C_{37}H_{38}O_6N_2$	711	$C_{42}H_{40}O_8Si_2$	456
$C_{37}H_{40}O_7N_2, 1\frac{1}{2}H_2O$	1392	$C_{42}H_{40}O_{12}Cr_2Sc_2$	1387
$C_{37}H_{42}O_2N_2$	711	$C_{42}H_{36}N_2Cl_6Br_2Pt$	67
C₃₈ Group.		$C_{42}H_{40}O_6Cl_6S_2Pt$	755
$C_{38}H_{34}O_8Cl_6Pt, 2H_2O$. 1107,	1150	$C_{42}H_{22}O_6N_2Cl_6Pt$	1800
$C_{38}H_{44}O_8N_2Cl_6Pt$ 1797, 1799		$C_{42}H_{36}O_{10}N_6S_2Cl_6Pt$	153
C₄₀ Group.		C₄₃ Group.	
$C_{40}H_{40}O_{10}N_2, 6H_2O$	710	$C_{43}H_{36}O_7NClBrS$	797
$C_{40}H_{46}O_{10}N_2$	710	C₄₄ Group.	
$C_{40}H_{46}O_{10}N_2, 4\frac{1}{2}H_2O$	710	$C_{44}H_{68}O_{20}N_8, H_2O$	1806
$C_{40}H_{48}O_{10}N_2$	709	$C_{44}H_{47}O_9N_4ClS, H_2O$	795,
$C_{40}H_{60}O_8As_2Cd$	2147	796	
$C_{40}H_{42}O_7N_6S_2Cr_2$	151	$C_{44}H_{80}O_7N_2S_2Si_2$	201, 203
$C_{40}H_{36}O_9N_2S_2Cr_2$	1696	$C_{44}H_{80}O_7N_2S_2Si_2, 4H_2O$. 468	
$C_{40}H_{44}O_7N_6Cl_6Pt$	343	C₄₆ Group.	
$C_{40}H_{46}O_6N_2Cl_6Pt$	1798	$C_{46}H_{68}O_7N_2S_2Si_2$	2094
$C_{40}H_{30}O_{10}N_6S_2Cl_6Pt$	151	$C_{46}H_{30}O_7N_2S_2Si_2$	2015
$C_{40}H_{36}O_2N_6Cl_6S_2Pt$	1696	$C_{46}H_{84}O_7N_2S_2Si_2$	2016
		$C_{46}H_{84}O_7N_2S_2Si_2, 4H_2O$	2016
		C₄₇ Group.	
		$C_{47}H_{44}O_7N_4, 6H_2O$	1223
		C₄₈ Group.	
		$C_{48}H_{34}O_6Cl_6PtSc_2$	1367
		$C_{48}H_{84}O_6Cl_6S_2Pt$	755, 758
		$C_{48}H_{54}O_{12}Cl_6S_2Pt$	757
		C₅₀ Group.	
		$C_{50}H_{84}O_{12}N_4, H_2O$	706
		$C_{50}H_{56}O_{12}N_6, 6H_2O$	706
		$C_{50}H_{50}O_{12}N_6, 7\frac{1}{2}H_2O$	706
		$C_{50}H_{58}O_{12}N_4, 7H_2O$	706
		C₅₄ Group.	
		$C_{54}H_{96}O_6Cl_6S_2Pt$	762
		C₅₈ Group.	
		$C_{58}H_{96}O_7$	1623
		C₆₃ Group.	
		$C_{63}H_{28}N_3Cl_3Br_3Au_2$	87

ERRATA.

VOL. LXV (TRANS., 1894).

Page Line
617 10* for "Trans., 1888, 53," read "Trans., 1887, 51, 610."

VOL. XCI (TRANS., 1907).

1641 2* "hydrochloric" read "acetic."
1642 19 "3H" read "3H."
1643 10* delete (second) "iodine."
1646 3* insert "+" before " k_1 ."
1647 8 for " K_1 " read " k_1 ."
1647 18 "ac" read " a_1 ."
1647 20 "a" read "A."
1647 1* "a" read "A."
1652 17 "0.000028" read "0.000058."
1652 11* In equation for " $(A-x)$ " read " $C(A-x)$."
1653 1* for "XII" read "XIII."
1658 19 "0.40" read "0.040."
1658 20 "0.535" read "0.0535."
1658 21 "0.83" read "0.083."
1658 2 (of table) for "0.5029" and "0.7368" read "0.4992" and "0.7301" respectively.

VOL. XCIII (TRANS., 1908).

49 5 for " $\frac{RT^2}{q}$ " read " $\frac{q}{RT^2}$."
49 14 "2860" read "6700."
258 5 "2CO₂" read "2C₂O₂."
486 1* " $C_{12}H_{14}O_{14}N_6 \cdot 3H_2O$ " read " $C_{12}H_{14}O_{14}N_6Mn \cdot 3H_2O$."
489 5 " $C_{12}H_{14}O_{14}N_6$ " read " $C_{12}H_{14}O_{14}N_6Ni$."
612 3* " $C_{20}H_{37}CO \cdot C_2H_5$ " read " $C_{20}H_{37}CO \cdot C_2H_3$."
849 3, 6 "23" read "21."
949 5 " d_{23} " read " d_{21} ."

1171 13* " C_6H_{10} $\begin{matrix} \text{CMe} \cdot \text{CO}_2\text{H} \\ \text{CO}_2\text{H} \\ \text{CH}_2 \cdot \text{NH}_2 \end{matrix}$ " read " C_6H_{10} $\begin{matrix} \text{CMe} \cdot \text{CO}_2\text{H} \\ \text{CO}_2\text{H} \\ \text{CH}_2 \cdot \text{NH}_2 \end{matrix}$."
1173 3 "standard" read "silver."

BONE AND COWARD'S PAPER.

1218 Table IX, 3rd column for "23.90" read "3.90."
1.40 21.40.

1219 "X, 6th" "71.9" read "28.1"
28.1 71.9

1222 "XII for Percentage composition of gaseous products. $\begin{cases} H_2 \\ C_2H_2 \\ C_2H_4 \\ C_2H_6 \\ CH_4 \end{cases}$ read Percentage composition of gaseous products. $\begin{cases} C_2H_2 \\ C_2H_4 \\ C_2H_6 \\ CH_4 \\ H_2 \end{cases}$

1404 13 for "pale yellow needles" read "almost colourless needles with only a slight yellow tinge."

1404 5* "pale yellow crystals" read "almost colourless crystals with only a slight yellow tinge."

1405 11 "p-Nitrotoluene" read "p-Nitrobenzaldehyde."

1836 6* "o-Chlorotoluene" read "o-Chlorobenzaldehyde."

1836 2* "p-Chlorotoluene" read "p-Chlorobenzaldehyde."

* From bottom.

Page	Line	
1687	8	for "m-Chlorotoluene" read "m-Chlorobenzaldehyde."
1798	3*	" $(C_{20}H_{25}O_4N_2)_2H_2PtCl_6$ " read " $(C_{20}H_{25}O_4N)_2H_2PtCl_6$."
1799	13*	" $(C_{19}H_{20}O_4N)_2H_2PtCl_6$ " read " $(C_{19}H_{20}O_4N)_2H_2PtCl_6$."
1899	16	" $C_{13}H_{18}O_4N_2As_2 \cdot 2\frac{1}{2}H_2O$ " read " $C_{13}H_{18}O_4N_2AsNa_2 \cdot 2\frac{1}{2}H_2O$."
2191	10	" $\frac{W \cdot K_2}{K_2 \cdot W}$ " read " $\frac{W' \cdot K_2}{K_2 \cdot W}$."
2192	4	after "H" insert " = ".
2192	21	delete "only."
2195	84	for " $\frac{K_2}{K_2} M$ " read " $\frac{K_2}{K_2} M$."
2197	18	should read " $S' = \frac{H^2 \cdot W''}{K' \cdot K_2 \cdot V^2} \left[2 \frac{M}{K_2} + 1 \right]$."
2197	18	for " $\frac{d^2(H^2)}{dA^2}$ " read " $\frac{d^2(H^2)}{dA^2}$."
2197	19	delete "providing . . . very great."
2197	20	delete "providing . . . very great."
2198	15	for "acid" read "acids."
2198	40	after "resembled" insert "the effect of water on catalysed."
2198	41	delete "by experiment."
2201	7	" "hydroxonium."
2203	3	before "value" insert "apparent" and delete " $\frac{\phi'}{\phi}$."

INDEX 1908.

2312 10 col ii. for "Methylcamphenonitrile" read "Methylcamphenonitrile."

* From bottom.

Organic Chemistry.

Syntheses of Methane by means of Calcium Hydride. M. MAYER and V. ALTMAYER (*Ber.*, 1908, 41, 3074—3080. Compare Abstr., 1907, i, 457).—Methane can be synthesised by the action of carbon, its monoxide, or dioxide on freshly-prepared calcium hydride. With carbon monoxide the reaction begins at 400°, and is rapid at 500°; thus when the gas is passed at the rate of 0.5 c.c. per minute the issuing mixture contains 42.75% methane and 51.86% hydrogen, and when the rate is 1 c.c. per minute the product contains over 60% of methane. At higher temperatures the amount of methane tends to decrease. The reaction can probably be represented by the equations: $3\text{CaH}_2 + 3\text{CO} = \text{CH}_4 + 3\text{CaO} + 2\text{C} + \text{H}_2$ and $\text{CaH}_2 + 2\text{C} = \text{CaC}_2 + \text{H}_2$.

Calcium carbide, calcium oxide, and carbon have been found in the residue. Carbon dioxide behaves in much the same manner as the monoxide.

With carbon (lamp-black) and calcium hydride, hydrogen is produced at 270°, and the formation of methane can only be recognised at higher temperatures. At 500° the gaseous mixture contains about 40% of methane.

J. J. S.

Studies in the Hexene and Heptene Series. NICOLAI D. ZELINSKY and E. S. PRISCHEVALSKY (*J. Russ. Phys. Chem. Soc.*, 1908, 40, 1105—1123).—The action of quinoline or of aqueous or alcoholic potassium hydroxide on *n*-hexyl iodide yields as main fraction a hydrocarbon, C_6H_{12} , b. p. 64—64.5°, D_4^{15} 0.6789, D_4^{20} 0.6750, n_D^{15} 1.3896, which gives only valeric acid on oxidation; the authors regard it as *n*-hexylene.

β -Methylhexyl iodide, $\text{C}_4\text{H}_9\cdot\text{CHMe}\cdot\text{CH}_2\text{I}$, prepared from the corresponding alcohol by the action of iodine and phosphorus, has b. p. 78—79°/19 mm., D_4^{17} 1.3707, D_4^{21} 1.3663, n_D^{21} 1.4891.

Methylpropylcyclopropane, $\text{CH}_2\begin{matrix} \text{CHMe} \\ \text{CHPr} \end{matrix}$, obtained by the action of alcoholic potassium hydroxide on *β -methylhexyl iodide*, b. p. 92—92.5°/753 mm., D_4^{15} 0.7077, D_4^{20} 0.7033, n_D^{15} 1.4044, gives, on oxidation with permanganate, (1) methyl *n*-butyl ketone; (2) butyric acid; (3) valeric acid; (4) heptic or *iso*heptic acid; (5) a glycol, $\text{C}_7\text{H}_{16}\text{O}_2$, possibly $\text{CH}_2\text{Pr}\cdot\text{CMe}(\text{OH})\cdot\text{CH}_2\cdot\text{OH}$, which, on further oxidation, gives butyric, valeric (?), and heptic or *iso*heptic acid. On adding hydrogen iodide to methylpropylcyclopropane and treating the resulting iodo-compound with dilute aqueous potassium hydroxide in a sealed tube at 100—105°, Δ^7 -heptylene, $\text{CHEt}\cdot\text{CHPr}$, b. p. 94.5—95.5°/757 mm., D_4^{15} 0.7124, D_4^{20} 0.7083, $n_D^{21.5}$ 1.4077, is obtained; on oxidation with permanganate, this hydrocarbon yields butyric and propionic acids, together with traces of acetone and valeric acid.

T. H. P.

Ozonides of the Simple Olefines. CARL D. HARRIES and KARL HAEFFNER (*Ber.*, 1908, 41, 3098—3102).—Although amylene and hexylene ignite when brought into contact with ozone, nevertheless it has been found possible to prepare their ozonides by using the method of Harries and Tank (this vol., i, 517), namely, treating a very dilute solution of the substance in an indifferent solvent with ozone. The ozonides of these olefines are very stable substances, and may be distilled in a vacuum. It is probable, from the observed values of the molecular refraction and dispersion of these ozonides, that the molecule contains one carbonyl oxygen atom and two ether oxygen atoms.

Amylene ozonide (*trimethylethylene ozonide*), $C_6H_{10}O_3$, is prepared by passing ozone into a dilute solution of the olefine in hexane, and heating at 60° in a vacuum the crude ozonide obtained after distilling off the solvent; it is a limpid, colourless liquid. The ozonide obtained by treating the crude ozonide with an aqueous solution of sodium hydrogen carbonate, when analysed, gives values which lie between those required for $C_6H_{10}O_3$ and $C_6H_{10}O_4$; it is far more explosive than the normal ozonide.

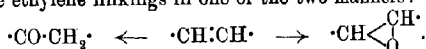
Hexylene ozonide, $C_6H_{12}O_3$, is prepared in the same way as the amylene ozonide. The crude ozonide first obtained is viscid, but yields the normal ozonide, a limpid liquid, when distilled at 60° under 12 mm. pressure. The amylene and hexylene ozonides are not readily decomposed by water.

Propylene ozonide is obtained by passing ozone into a dilute solution of propylene in ethyl chloride cooled in a carbon dioxide-ether freezing mixture. It is a limpid liquid with a pungent odour, b. p. $29-30^\circ/19.5$ mm., becomes solid at low temperatures, explodes with great violence when heated in a test-tube, and is rapidly decomposed by water.

W. H. G.

Absorption of Oxygen by Electro-condensation Products. SIMA M. LOSANITSCH (*Monatsh.*, 1908, 29, 753—762).—Chiefly an account of work already published (*Abstr.*, 1897, i, 179; this vol., ii, 32), and a criticism of Jovitschitsch's work (this vol., i, 118).

Since the compound $(C_2H_2O)_n$, formed by absorption of oxygen by the electro-condensation product of ethylene, distils unchanged, the oxygen must be in chemical combination. The addition may take place at the ethylene linkings in one of the two manners:



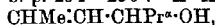
The solid condensation product of acetylene, when treated with ozonised oxygen, forms a compound, $(C_6H_8O_2)_n$; the liquid condensation product, on the other hand, absorbs oxygen only slowly, and, after several months, contains less oxygen than required for the formula $(C_6H_{10}O)_2$. Both condensation products of acetylene with ethylene absorb oxygen, but without forming definite compounds. The oxygenated compound derived from acetylene and carbon monoxide absorbs more oxygen than previously stated, but still without reaching the composition $(C_3H_3 \cdot CO \cdot O_2)_n$.

G. Y.

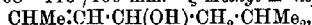
Action of Organo-magnesium Compounds on Crotonaldehyde and the Optical Behaviour of the Products. JOHANN REIF (*Ber.*, 1908, 41, 2739—2746).—The paper is a continuation of Triguard's research on the action of magnesium methyl iodide or magnesium isomyl bromide on crotonaldehyde (*Abstr.*, 1901, i, 679). Employing the same method, the author has examined the action of magnesium ethyl, propyl, isopropyl, and isobutyl bromides. The resulting unsaturated alcohols, $\text{CHMe}:\text{CH}:\text{CH}(\text{OH})\cdot\text{CH}_2\text{R}$, described below are colourless, mobile liquids, which exhibit a normal molecular refraction, are oxidised by 1% potassium permanganate to the corresponding glycerols, and by distillation with potassium hydrogen sulphate yield diolefines of the type $\text{CHMe}:\text{CH}:\text{CH}:\text{CHR}$, which, in agreement with Brühl's observations on substances containing the group $>\text{C}:\text{CH}:\text{CH}:\text{C}<$, exhibit a marked exaltation of the molecular refraction.

The following new compounds are mentioned:

Δ^3 -Pentene- δ -ol yields a *chloride*, $\text{CHMe}:\text{CH}:\text{CHMeCl}$, b. p. 103—106° (decomp.), and a *glycerol*, $\text{C}_5\text{H}_{12}\text{O}_3$, b. p. 244—246°, of which the *triacetate* has m. p. 121° and b. p. 241—243°. Δ^3 -Hexene- δ -ol, $\text{CHMe}:\text{CH}:\text{CHEt}:\text{OH}$, b. p. 133—135° or 85—87°/118 mm., D_4^{20} 0.8409, n_D^{25} 1.4312; the *acetate* has b. p. 153—155°; the *chloride* has b. p. 122—126° (decomp.) or 65—70°/110 mm., and is converted by methyl-alcoholic potassium hydroxide into δ -methoxy- Δ^3 -hexene, $\text{CHMe}:\text{CH}:\text{CHEt}:\text{OMe}$, b. p. 110—113° (compare Vaubel, *Abstr.*, 1891, 996); the *glycerol*, $\beta\gamma\delta$ -hexantriol, has b. p. 256—257°, and yields a solid *triacetate*, b. p. 254—256°. Δ^3 -Heptene- δ -ol,

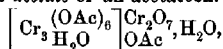


b. p. 152—154° or 104—105°/106 mm., D_4^{20} 0.8445, n_D^{25} 1.4373, forms an *acetate*, b. p. 168—170°, and a *chloride*, b. p. 140—144° or 36—90°/104 mm. ζ -Methyl- Δ^3 -hexene- δ -ol, $\text{CHMe}:\text{CH}:\text{CHPr}^n:\text{OH}$, has b. p. 139—140° or 92—94°/105 mm., D_4^{20} 0.8426, n_D^{25} 1.438; the *acetate* has b. p. 108—110°/100 mm. ζ -Methyl- Δ^3 -heptene- δ -ol,



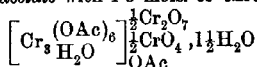
D_4^{20} 0.8389, n_D^{25} 1.4395, forms an *acetate*, b. p. 123—126°/108 mm. Δ^3 -Pentadiene, C_5H_8 , b. p. 40—41°, D_4^{25} 0.6794, n_D^{25} 1.4206. Δ^3 -Hexadiene, C_6H_{10} , b. p. 80—82°, D_4^{25} 0.7177, n_D^{25} 1.4463, yields a *dihydrobromide*, b. p. 199—201° or 98—102°/30 mm., and a *tetrabromide*, n. p. 180°. Δ^3 -Heptadiene, C_7H_{12} , b. p. 104—106°, D_4^{25} 0.7327, n_D^{25} 1.4486. ϵ -Methyl- Δ^3 -hexadiene, C_8H_{14} , b. p. 97—99°, D_4^{25} 0.7192, n_D^{25} 1.4266. ζ -Methyl- Δ^3 -heptadiene, C_8H_{14} , b. p. 114—116°, D_4^{25} 0.7401, n_D^{25} 1.4397. η -Methyl- Δ^3 -octadiene, C_9H_{16} , b. p. 149°, D_4^{25} 0.7521, n_D^{25} 1.4543, yields a *dihydrobromide*, b. p. 124—126°/16 mm., and a *tetrabromide*, b. p. 184°/18 mm. C. S.

Salts of an Acetatochromo-base. RUDOLF F. WEINLAND (*Ber.*, 1908, 41, 3236—3245).—When chromic acid (1 part) is warmed at 100° with glacial acetic acid (2 parts) until no more carbon dioxide is evolved, the *dichromate acetate* of an acetatochromo-base,



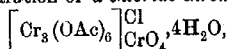
is obtained in brownish-black, microscopic prisms. If this salt is

treated with a little water, it first dissolves, and afterwards olive-green plates of a *chromate acetate* with 1.5 mols. of chromic acid,



are precipitated. On the other hand, if the dichromate acetate is recrystallised from a larger quantity of water, dark green crystals of a simpler *chromate acetate*, $\left[\text{Cr}_3 \begin{pmatrix} (\text{OAc})_6 \\ \text{H}_2\text{O} \end{pmatrix} \right] \begin{pmatrix} \text{CrO}_4, 3\frac{1}{2} \text{H}_2\text{O} \\ \text{OAc} \end{pmatrix}$, are produced. This compound is also formed directly from the two acids.

Addition of hydrochloric acid to a solution of the dichromate acetate causes the separation of a *chloride chromate*,

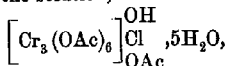


as dark green crystals.

The *platinichloride*, $\left[\text{Cr}_3 (\text{OAc})_6 \right] \begin{pmatrix} (\text{OH})_2 \\ \frac{1}{2} \text{PtCl}_6, 5\text{H}_2\text{O} \end{pmatrix}$, is obtained from all the above salts of the base.

A basic *chromate acetate*, $\left[\text{Cr}_3 (\text{OAc})_6 \right] \begin{pmatrix} \text{OH} \\ \frac{1}{2} \text{CrO}_4, 5\text{H}_2\text{O} \\ \text{OAc} \end{pmatrix}$, is obtained when

lead acetate is added to the dichromate acetate until only $\frac{1}{4}$ mol. of chromic acid is left in the solution, and a basic *chloride*,



and basic *nitrate*, $\left[\text{Cr}_3 (\text{OAc})_6 \right] \begin{pmatrix} \text{OH} \\ \text{NO}_3, 2\frac{1}{2} \text{H}_2\text{O} \\ \text{OAc} \end{pmatrix}$, can be obtained by similar

means. The *diacetate*, $\left[\text{Cr}_3 (\text{OAc})_6 \right] \begin{pmatrix} \text{OH} \\ (\text{OAc})_2, 5\text{H}_2\text{O} \end{pmatrix}$, is obtained from the solution of the chromate acetate, after treatment with lead acetate, as a light green, crystalline powder. Silver nitrate precipitates silver acetate from its concentrated solution, proving the presence of acetic acid ions, but the nitrate acetate solution gives no such precipitate. The method of preparation of these compounds shows that the positive complex contains three atoms of chromium and six acetic residues. The water remaining after drying to constant weight in a vacuum over sulphuric acid is held to be joined to the cation.

The free acetatochromo-base prepared from the chloride acetate and excess of silver oxide is dark green in solution. The alkaline liquid is unstable, and gradually becomes acid.

The corresponding *iron base*, $\left[\text{Fe}_3 (\text{OAc})_6 \right] \begin{pmatrix} \text{Cr}_2\text{O}_7, \text{H}_2\text{O} \\ \text{OAc} \end{pmatrix}$, and propionato-bases have been prepared. W. R.

Stereochemical Nature of the Addition of Chlorine to Crotonic Acid. ARTHUR MICHAEL and O. D. E. BUNGE (*Ber.*, 1908, 41, 2907—2913).—Ordinary fumaroid crotonic acid absorbs chlorine both in the dark and in sunlight, forming $\alpha\beta$ -dichlorobutyric acid, m. p. 63°, and the same compound is obtained under similar conditions

from *allocrotonic acid*. A stereoisomeric dichlorobutyric acid, m. p. 78° , is produced by the addition of hydrogen chloride to α -chloro-crotonic acid, and is regarded as the maleoid derivative. That this is not the case is shown by the fact that the more fusible dichlorobutyric acid has a greater electrical conductivity than the isomeride, and it must therefore be regarded as the maleoid derivative (crotonic acid *allodichloride*). The fumaroid acid yields, primarily, maleoid additive products, and the further isomeric change takes place much less readily in the case of the crotonic acids than with the cinnamic acids. Actinic rays are unable to effect it, and it requires prolonged heating with concentrated hydrochloric acid to bring about the conversion of crotonic acid *allodichloride* into the less fusible isomeride.

Of the two dichlorosuccinic acids melting at 175° and 215° , the more fusible isomeride is the stronger acid.

E. F. A.

Action of Ozone on Compounds containing Double and Treble Linkings. ETTORE MOLINARI (*Ber.*, 1908, 41, 2782—2785).—The author replies to Harries' criticisms (this vol., i, 387), and re-states his right to work on ozonides.

C. S.

Triolein Ozonide and its Decomposition Products. ETTORE MOLINARI and P. FENAROLI (*Ber.*, 1908, 41, 2789—2794).—When triolein is ozonised in hexane solution, an insoluble, gelatinous, yellow mass, probably a polymerised ozonide, is obtained, which is converted by thirty minutes' heating with 1% potassium hydroxide on the water-bath into *triolein ozonide*, $C_{57}H_{104}O_{15}$, a viscous, colourless oil, which decomposes at 136° . This ozonide, not the polymerised form, is obtained by the ozonisation of olive oil. It is decomposed by 30% alcoholic potassium hydroxide on the water-bath, yielding glycerol, uzelaic and nonoic acids, a white hydroxy-acid, $C_{18}H_{34}O_3$ (compare Molinari and Soncini, *Abstr.*, 1906, i, 792), and an oily acid, $C_{18}H_{32}O_6$, which is oxidised quantitatively to azelaic acid by potassium permanganate. Hydrogen peroxide is not formed, and aldehydes only in small amount.

Arnold and Mentzel's benzidine reaction is the best test for hydrogen peroxide in the presence of aldehydes; some of the higher aldehydes give with potassium ferricyanide and ferric chloride, or with titanium sulphate, the same reactions as hydrogen peroxide.

The authors hope that the saponification value of ozonides will form a new constant in the analysis of oils and fats.

U. S.

Products of the Decomposition of Oleic Acid Ozonide. ETTORE MOLINARI and C. BAROSI (*Ber.*, 1908, 41, 2794—2799).—According to Harries, the primary products of the decomposition of oleic acid ozonide by hot water are exclusively hydrogen peroxide, ozonaldehyde, and the semialdehyde of azelaic acid (*Abstr.*, 1906, i, 793; 907, i, 11); Molinari and Soncini (*Abstr.*, 1906, i, 792; compare also preceding abstract) obtained no hydrogen peroxide, but aldehydes in small amount, and mainly azelaic, nonoic, and two other acids, $C_{18}H_{32}O_6$ and $C_{18}H_{34}O_3$. To ascertain whether these four acids are

secondary oxidation products, the authors have investigated the decomposition of oleic acid ozonide, D¹⁸1·0218, by hot saturated potassium hydrogen sulphite. The reaction is very complicated, and several of the products have not yet been investigated. The main results are: (a) primarily formed acids are azelaic, nonoic, and the acids $C_{18}H_{32}O_6$ and $C_{18}H_{30}O_8$. The acid $C_{18}H_{30}O_3$ (calcium salt insoluble) has m. p. 41° (compare Molinari and Soncini, *loc. cit.*), and is a hydroxystearic acid, $CH_3 \cdot [CH_2]_{17} \cdot CH(OH) \cdot [CH_2]_3 \cdot CO_2H$, produced by the aldol condensation of nonaldehyde and nonoic acid. The acid $C_{18}H_{32}O_6$ (calcium salt soluble) is probably produced by the condensation of 2 mols. of the semialdehyde of azelaic acid; (b) the aldehydes liberated from their bisulphite compounds undergo polymerisation; *paranonaldehyde*, $(C_9H_{18}O)_3$, a white, crystalline mass, m. p. 28°, has been isolated, and also another substance, b. p. 190°, which forms a *semicarbazone*, $C_{19}H_{30}O_3N_2$, m. p. 54°, and probably is formed by the aldol condensation of 2 mols. of nonaldehyde; (c) the semialdehydes are isolated in the form of a viscous, brown oil, which no longer shows the properties of aldehydes, and consists probably of condensation or oxidation products of the semialdehyde of azelaic acid. By prolonged boiling with water, azelaic acid is extracted, and the residue contains the acid $C_{18}H_{30}O_6$ and other unexamined substances. C. S.

Constitution of the So-called Elæomargaric Acid. TOKUHEI KAMETAKA (*J. Coll. Sci. Tôkyô*, 1908, 25, iii, 1—8. Compare *Trans.*, 1903, 83, 1042).—Elæomargaric acid cannot be a homologue of sorbic acid, as stated by Cloez (this *Journ.*, 1877, i, 454), since it does not yield tartaric acid when oxidised with potassium permanganate (compare Doebner, *Abstr.*, 1890, 1274). Cloez also states that it is a homologue of stearolic acid, but this is improbable, since it does not give a definite product with concentrated sulphuric acid. As stated by Maquenne (*Abstr.*, 1903, i, 62), elæomargaric acid when oxidised yields azelaic acid, but the presence of valeric acid could not be established. It is therefore highly probable that one of the two ethylene linkings in elæomargaric acid is in the middle of the chain of eighteen carbon atoms. The other ethylene linking is probably situated on that side of the middle ethylene linking which is further removed from the carboxyl group. This view is supported by the behaviour of the acid towards aniline; it yields an *anilide*, $C_{18}H_{31}O \cdot NHPh$, which crystallises in lustrous scales, m. p. about 80°, but does not form an aniline additive product; consequently, an ethylene linking is not near the carboxyl group (compare Autenrieth and Pretzell, *Abstr.*, 1903, i, 474). The anilide combines with bromine, forming a *substance*, m. p. about 95°.

The *ethyl* ester of elæomargaric acid is a yellow liquid, b. p. 230—240°/25 mm; the *methyl* ester decomposes when warmed.

W. H. G.

Japanese Vegetable Oils. TOKUHEI KAMETAKA (*J. Coll. Sci. Tôkyô*, 1908, 25, iv, 1—7).—*Japanese Wood Oil (Kiri-abura)*.—The iodine number of the fresh, cold-drawn oil was found by Hübl's method to be 161·3; after storing in a stoppered bottle in the dark for eleven

months, the value obtained by the same method was 150.0. The iodine number after three months, determined by Wys' method, was 156.3, and after eleven months, 153.2. The low iodine number of Japanese wood oil cannot, therefore, be ascribed to previous oxidation, as stated by some chemists (compare Ingle, *J. Soc. Chem. Ind.*, 1902, 21, 187), although the value does decrease on oxidation. Using the number 161.3, it is found by calculation that the oil consists of about 86% of elaeomargarin and 14% of olein. The following constants were determined: acid number, 3.81; D_{25}^{20} 0.9307; saponification number, 196.0. An enzyme, capable of hydrolysing the oil and also ethyl acetate, was obtained from the seeds of *Elaeococca vernicia*. Since this enzyme is present in small quantities in the oil itself, it possibly plays some part in the rapid drying of this oil.

Camellia Oil or Oil of *Thea japonica* (*Tsubaki-abura*) has D_{25}^{20} 0.9138 and iodine number (Hübl's method) 79.47. The principal acid in the oil is oleic acid.

Oil of *Terrya nucifera* (*Kayana-abura*) has the iodine number (Hübl's method) 137.3. It contains linolic acid or its isomerides, together with stearic acid.

Soya-bean Oil or Oil of *Glycine hispida* (*Daizyu-abura*).—The unsaturated acids in this oil are chiefly oleic acid and linolic acid or its isomerides.

Oil of *Perilla* (*Eno-abura*) appears to contain linolenic acid or its isomeride.

W. H. G.

Compounds of Bismuth with Aliphatic Hydroxy-acids.
HANS TELLE (*Arch. Pharm.*, 1908, 246, 484—503).—Compounds of bismuth with lactic, malic, tartaric, and citric acids have been prepared, and a new method of preparing such salts, depending on the decomposition of the soluble hydrated acid lactate with the appropriate acid, is described.

Freshly-precipitated bismuth hydroxide dissolves slowly in lactic acid slightly diluted with water, and the solution gradually deposits bunches of glancing needles of the hydrated salt, $\text{BiC}_6\text{H}_9\text{O}_6 \cdot 7\text{H}_2\text{O}$. This is readily soluble in water, and at 105° furnishes the anhydrous salt, $\text{BiC}_6\text{H}_9\text{O}_6$, which is scarcely soluble in water, and can be obtained in rhombic tablets by digesting bismuth hydroxide with lactic acid at 100°. The anhydrous salt decomposes slightly when boiled for some time with water (compare Engelhardt, this Journ., 1848, 1, 400, and Brunig, *Annalen*, 1857, 104, 195).

Bismuth malate, $\text{BiC}_4\text{H}_5\text{O}_6 \cdot \text{H}_2\text{O}$, colourless needles, can be prepared either by heating bismuth hydroxide with just enough malic acid dissolved in a little water for a few minutes, or by adding a solution of malic acid to one of the hydrated lactate referred to above and boiling the mixture. It is nearly insoluble in water.

Attempts to prepare bismuthotartaric acid by Baudran's method (*Abstr.*, 1900, i, 375) resulted in the formation of minute needles of the hydrated salt, $\text{BiC}_2\text{H}_3\text{O}_4 \cdot 3\text{H}_2\text{O}$, instead of the compound, $\text{C}_2\text{H}_3(\text{BiO})\text{O}_6 \cdot \text{H}_2\text{O}$, described by Baudran (compare Rosenheim and Vogelsang, *Abstr.*, 1906, i, 231). This can also be obtained by adding a solution of tartaric acid to one of the hydrated lactate and boiling

the liquid. It is nearly insoluble in water, but dissolves in solutions of alkali hydroxides or carbonates, yielding syrups from which no crystalline product could be obtained.

Rother's bismuth citrate, $\text{BiC}_6\text{H}_5\text{O}_7$ (this Journ., 1876, ii, 173), can be prepared in a well-crystallised condition, either by dissolving bismuth hydroxide in a solution of citric acid, or from the hydrated lactate by the general method. T. A. H.

Dehydrocamphenylic Acid. GUSTAV KOMPPA and S. V. HINTIKKA (*Ber.*, 1908, 41, 2747—2750).—By the oxidation of camphene by dilute nitric acid, Jagelki obtained an unsaturated acid, $\text{C}_{10}\text{H}_{14}\text{O}_2$, which he claimed to be identical with Wagner's dehydrocamphenylic acid (Abstr., 1899, i, 627, 629). Since the latter is saturated, the authors have re-examined Jagelki's compound, and find that it is saturated and identical with dehydrocamphenylic acid and with an acid obtained by Konowaloff (Abstr., 1907, i, 279). The *silver*, *lead*, and *calcium* salts are mentioned. The *ethyl* ester, $\text{C}_{12}\text{H}_{18}\text{O}_2$, b. p. 100—101°/10 mm., D_{20}^{20} 1.0143, n_D^{20} 1.47299, shows the molecular refraction of a saturated, tricyclic ester. The *amide*, m. p. 114.5°, and *anilide*, $\text{C}_9\text{H}_{13}\text{CO-NHPh}$, m. p. 102—103°, have been prepared. C. S.

Active Pinonic and Pinic Acids. PHILLIP BARBIER and VICTOR GRIGNARD (*Compt. rend.*, 1908, 147, 597—600).—Previous authors, using pinene of low rotatory power, have only succeeded in obtaining racemic pinonic acid by oxidation of this terpene. When *l*-pinene (b. p. 155—157°, $[\alpha]_D^{25}$ -37.2°) is oxidised by potassium permanganate in the cold, a mixture of racemic pinonic acid and *l*-pinonic acid is produced. *l*-Pinonic acid, $\text{C}_{10}\text{H}_{16}\text{O}_3$, forms large crystals, m. p. 67—69°, $[\alpha]_D^{25}$ -90.5° (in chloroform), and gives a mixture of two *oximes*, the γ crystallising in dextrorotatory micro-crystals, m. p. 189—191°, difficultly soluble in ether, and the β crystallising in large, levorotatory tablets, m. p. 128°, readily soluble in ether. On oxidation by sodium hypochlorite or hypobromite, the acid gives *d*-pinic acid, having m. p. 135—136°, $[\alpha]_D^{25}$ +7.1°. A *d*-pinene, having D_0 0.8745, D_4 0.8635, n_D 1.46977, $[\alpha]_D^{25}$ +39.4°, b. p. 155—158°, on oxidation in a similar manner, gives a mixture of *d*-pinonic acid and the racemic acid. *d*-Pinonic acid has m. p. 67—68°, $[\alpha]_D^{25}$ +89.0°, and, when fused with an equal weight of the *l*-acid, gives immediately the racemic acid, m. p. 104°. The *d*-acid gives the β and γ oximes identical with those described by Baeyer. The conclusion is drawn that Tiemann's acid, having m. p. 98—99°, and giving an oxime, m. p. 147°, is not the true *l*-pinonic acid derived from *l*-pinene; the two acids are possibly the *cis*- and *cis-trans*-isomerides predicted by theory. E. H.

Equilibrium in the System: Potassium Oxalate, Oxalic Acid, Water. IWAN KOPPEL and M. CAHN (*Zeitsch. anorg. Chem.*, 1908, 60, 53—112).—The equilibrium relations in the system $\text{K}_2\text{O}-\text{CrO}_3-\text{H}_2\text{O}$ have been determined over a wide range of temperature, and the results are illustrated by numerous curves and are summarised on projected space diagrams. The three potassium oxalates already known, $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, KHC_2O_4 , and $\text{KHC}_2\text{O}_4 \cdot \text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$,

exist in equilibrium with solution within certain limits of concentration at all the temperatures at which measurements have been made. Further, an acid salt of the formula $2K_2C_2O_4 \cdot H_2C_2O_4 \cdot 2H_2O$ (the salt *N*), not previously known, is stable between its cryohydric point and its transition point in the neighbourhood of 84° .

In the course of the investigation, the complete isothermals have been determined at 0° , 30° , and 60° by solubility observations in the usual way, and the ice lines and boiling-point lines have also been determined. The *M*-salt is decomposed by water under all conditions, with separation of potassium hydrogen oxalate, and this salt is decomposed by water from its cryohydric point up to about 50° . Oxalic acid itself is partly decomposed when heated in aqueous solution above 100° , even in the presence of acid oxalates.

The transition temperature of the *M*-salt, represented by the equation: $2K_2C_2O_4 \cdot H_2C_2O_4 \cdot 2H_2O \rightleftharpoons K_2C_2O_4 \cdot H_2O + 2KHC_2O_4 + H_2O$, was determined both by solubility and dilatometer experiments, the mean value being about 84° , as mentioned above.

In preparing pure potassium hydrogen oxalate, rather more than the theoretical amount of alkali should be employed, otherwise the salt which separates from solution is contaminated with tetroxalate. Observations have been made which support Wyruboff's statement, that the acid oxalate exists in two forms: an anhydrous modification in monoclinic crystals, stable above 15° , and a monohydrate in orthorhombic crystals, which separates from solution below 15° . G. S.

Decomposition of Lead Oxalate by Saline Solutions.

H. CANTONI and L. MAURI (*Bull. Soc. chim.*, 1908, [iv], 3, 929—935).—An extension of the investigation previously described (*Abstr.*, 1906, i, 557) on the action of alkali sulphates on oxalates of the alkaline earths to the case of lead oxalate reacting with solutions of alkali chlorides or sulphates. Tables of the numerical results obtained are given in detail in the original. These show that alkali sulphates in solution decompose lead oxalate, and that the extent of this decomposition increases (1) with rise of temperature, (2) time of action, and (3) concentration of the solution. Of the three sulphates tried, namely, potassium, sodium, and ammonium, the first is the most active and the third least, but the potassium salt is relatively much more active than either of the other two. The alkali chlorides attack the oxalate far less readily than do the sulphates, and there is little to choose between the three chlorides tried in this respect. In the cold, neither alkali sulphates nor chlorides exert any considerable action on lead oxalate. T. A. H.

Complex Ferrimalonates. MOTOOKI MATSUI (*J. Coll. Sci. Tôkyô*, 1908, 25, ii, 1—5).—Complex ferrimalonates having the composition $(CO_2M \cdot CH_2 \cdot CO_2)_3Fe$, where *M* represents either sodium, potassium, or ammonium, have been prepared by adding ferric chloride to an aqueous solution of the malonate so long as the mixed solution did not give a red coloration with potassium thiocyanate. These salts are decomposed by acids and alkalis; in aqueous solution, they dissociate into the complex ion, $[Fe(CO_2 \cdot CH_2 \cdot CO_2)_3]^{3-}$, and the ion of the alkali

metal. The aqueous solution is unstable, the green colour soon changing into brown; it is probable that the decomposition is due to hydrolysis, since the addition of sodium malonate renders the solution stable. The acid corresponding with these salts has not yet been isolated, but it is obtained in aqueous solution by dissolving moist ferric hydroxide in a warm solution of malonic acid.

Sodium ferrimalonate, $\text{Fe}(\text{CO}_2\cdot\text{CH}_2\cdot\text{CO}_2\cdot\text{Na})_3$, forms green, sandy crystals and decomposes at about 200° . *Potassium ferrimalonate*, $\text{Fe}(\text{CO}_2\cdot\text{CH}_2\cdot\text{CO}_2\cdot\text{K})_3\cdot 4\text{H}_2\text{O}$, crystallises in green prisms. *Ammonium ferrimalonate*, $\text{Fe}(\text{CO}_2\cdot\text{CH}_2\cdot\text{CO}_2\cdot\text{NH}_4)_3\cdot 4\text{H}_2\text{O}$, forms green, prismatic crystals. The *barium* and *calcium* salts were prepared, but not analysed.

Ferrous iron also forms complex malonates, which are precipitated as yellow crystals by adding alcohol to an aqueous solution of a malonate and ferrous sulphate. They are very unstable, and immediately change in the air to the corresponding ferric compounds.

W. H. G.

Formation of Compounds in Solutions of Tartaric Acid and Sodium Molybdate. HERMANN GROSSMANN (*Zeitsch. anorg. Chem.*, 1908, 60, 50—52).—Remarks on a recent paper by Quinet (this vol., i, 713). Priority is claimed for certain results described in Quinet's paper (Rosenheim and Itzig, *Abstr.*, 1900, i, 135, 272; Grossmann and Pötter, 1906, i, 799). Further, it is pointed out that the mixture of tartaric acid and sodium molybdate of maximum rotatory power contains the components in the ratio $1\text{C}_4\text{H}_6\text{O}_6 : 1\text{Na}_2\text{MoO}_4$, and not 1:2 as given by Quinet, and that the other characteristic point corresponds with the composition $\text{C}_4\text{H}_6\text{O}_6 : \frac{1}{2}\text{Na}_2\text{MoO}_4$. G. S.

Some Citrates. E. I. VAN ITALLIE (*Pharm. Weekblad*, 1908, 45, 1201—1210).—The action of ammonia on aqueous solutions of citric acid at 30° only yields known hydrates; that of calcium hydroxide at the same temperature produces the hydrates $(\text{C}_6\text{H}_7\text{O}_7)_2\text{Ca}\cdot 3\text{H}_2\text{O}$ and $\text{C}_6\text{H}_6\text{O}_7\cdot\text{Ca}\cdot 4\text{H}_2\text{O}$. A. J. W.

Action of Calcium Carbide on Some Ketones. F. BOBROUX and FELIX TABOURY (*Bull. Soc. chim.*, 1908, [iv], 3, 829—833).—Acetone prepared from its sodium hydrogen sulphite compound reacts energetically with powdered calcium carbide, evolving acetylene and forming mesityl oxide and more complex substances boiling between 170° and 350° . Whatever the conditions, the proportion of mesityl oxide only varies from 8% to 12%; the yield of the higher boiling products, on the other hand, depends on the length of time the acetone is in contact with the carbide. Commercial acetone, the vapour of which acts on fragments of calcium carbide, has no action on the latter when powdered. Pure butanone also acts on calcium carbide, although more slowly, giving, besides higher boiling products, an agreeably smelling ketone, $\text{C}_8\text{H}_{14}\text{O}$, b. p. 164 — $166^\circ/760$ mm. and 66 — $68^\circ/20$ mm., D^{20}_D 0.853. This is probably either ζ -methyl- Δ^2 -heptene- γ -one, $\text{CH}_3\text{Me}\cdot\text{CO}\cdot\text{CH}:\text{CMe}\cdot\text{CH}_2\text{Me}$, or $\gamma\delta$ -dimethyl- Δ^2 -hexene- β -one, $\text{CH}_3\text{Me}\cdot\text{CMe}\cdot\text{CMe}\cdot\text{CO}\cdot\text{Me}$, or a mixture

of the two. If commercial butanone, dried over potassium carbonate and rectified, is used, the new ketone apparently contains a liquid richer in oxygen.

Boiling δ -pentanone and acetophenone have no action on calcium carbide. The vapour of the latter ketone at ordinary, but not at reduced, pressure acts on the carbide, giving a gummy mass.

The author finds that certain esters retard or altogether prevent the action of acetone, and draws the conclusion that the inactivity of commercial acetone is due to its containing an impurity of this nature.

E. H.

Reduction of $\alpha\beta$ -Unsaturated Ketones. ALADAR SKITA, A. ARDAN, and M. KRAUSS (*Ber.*, 1908, 41, 2938—2946).—The paper deals with the comparative study of the reduction of aliphatic and alicyclic $\alpha\beta$ -unsaturated ketones by Sabatier and Senderens' method.

Mesityl oxide, reduced at 185° for seven hours, yields methyl isobutyl ketone in 60% yield and a small quantity of methylisobutylcarbinol; reduced for eight hours at 210° in a more rapid current of hydrogen (220 c.c. per minute), mesityl oxide yields β -methylpentane (compare Darzens, *Abstr.*, 1905, i, 66, 172). Phorone, reduced at 225° for five hours (velocity of hydrogen = 100 c.c. per minute), gives a 65% yield of valerone, whilst at 235° and with a velocity of 280 c.c. of hydrogen per minute, the products are valerone and an unidentified, unsaturated hydrocarbon, diisobutylcarbinol, and $\beta\zeta$ -dimethylheptane.

1:3-Dimethyl- Δ^3 -cyclohexen-5-one, reduced for six hours at 235° (velocity of hydrogen = 100 c.c. per minute), yields 1:3-dimethylcyclohexane, b. p. 119 — 120° , D_{20}^{20} 0.7822. Ethyl 1-methyl- Δ^2 -cyclohexen-5-one-2-carboxylate (Hagemann, *Abstr.*, 1893, i, 393), reduced for four hours at 280 — 285° with an equally rapid current of hydrogen, yields ethyl 1-methyl- Δ^4 (or Δ^3)-cyclohexene-2-carboxylate, of which the acid has b. p. 114 — $130^\circ/9$ mm., and forms a crystalline dibromide, $C_8H_{12}O_4Br_2$. C. S.

Density of Sucrose. G. FOUQUET (*Bull. Assoc. Chim. Sucr. Dist.*, 1908, 26, 176—177).—The author finds that different methods of crystallising sucrose produce crystals of varying density; one specimen of crystals examined had D 1.550 to 1.575, whilst another had D 1.580 to 1.610. The actual cause which produces the variation in the density has not yet been ascertained.

W. P. S.

Aqueous Solutions and their Densities: Sucrose Solutions. G. FOUQUET (*Bull. Assoc. Chim. Sucr. Dist.*, 1908, 26, 160—176).—The curve of contractions occurring when sucrose is dissolved in water, calculated on the value 1.590 for D_{15}^{15} of sucrose, exhibits a maximum for 40% by weight of sucrose, whilst for a saturated solution (66—67% of sucrose) the contraction is zero. The above value for the density of sucrose is calculated from the value D_{15}^{15} 1.5881. For concentrations up to 15%, the contraction is sensibly proportional to the concentration, whilst for concentrations (p) up to 40%, the contraction is represented by the formula: $\delta = 0.000225p(80 - p)$. When p increases from 5% to 75%, the density of the sugar in solution diminishes continuously, and the same is probably the case for increase of p from 0% to 5%.

although here the increase is only slight. For concentrations up to 5%, the values of D_1^{25} for sucrose solutions are represented by the expression: $162.91956/(163.05 - 0.6313 p)$. These considerations are extended to impure sucrose solutions.

T. H. P.

Inversion of Sucrose. YUKICHI OSAKA (*J. Coll. Sci. Tokyo*, 1908, 25, i, 1—8).—It is shown that, whereas in moderately dilute solutions the hydrolysis of sucrose by an acid may be regarded as practically complete, in strong solutions (80 grams of sugar in 100 c.c. of solution) the hydrolysis is incomplete. It is therefore probable that the inversion of sucrose by an acid is a reversible reaction. This view is supported by the result of the following experiment. A solution of about 20 grams of sucrose in 50 c.c. of solution was hydrolysed completely by hydrochloric acid; 5.7507 grams of this solution, treated with sodium acetate and diluted to 25 c.c., gave a rotation of -3.83° . The same weight of the inverted solution (5.7507 grams) was then concentrated to about one-half by passing dry air over the surface of the solution; it was then treated with sodium acetate and made up to 25 c.c. as before; this solution had a rotation of -3.30° . The reversion product could not be maltose or isomaltose, since the author finds that no reversion takes place in concentrated solutions of dextrose under the influence of an acid at 25° during a period of two months. W. H. G.

Inversion of Sucrose by Invertase. II. C. S. HUDSON (*J. Amer. Chem. Soc.*, 1908, 30, 1564—1583).—A continuation of the work previously recorded (this vol., i, 605). The results completely confirm O'Sullivan and Tompson's conclusions (*Trans.*, 1890, 51, 834).

The velocities of the mutarotation of dextrose and laevulose at 30° in water and in aqueous solutions of hydrochloric acid and invertase have been determined. The velocities are not affected by invertase, but are greatly accelerated by acids. This difference between the action of invertase and acids on the products of the inversion of sucrose causes the action of invertase to appear irregular, owing to the influence of the mutarotation of the invert-sugar on the polarimetric reading. On correcting for this, the inversion of sucrose by invertase proves to be a catalytic reaction of the first order.

The acceleration of the mutarotation of dextrose at 30° by hydrochloric acid is such that the rate is a linear function of the hydrogen-ion concentration. In the case of laevulose, a minimum rate occurs in dilute acid solutions similar to the less pronounced minimum which has been found in the case of dextrose at 25° (*Abstr.*, 1907, ii, 942).

The action of invertase is greatly accelerated by minute traces of acid, but the addition of further small quantities of acid does not increase the effect. The inversion of sucrose, in both dilute and concentrated solutions, by invertase is proportional to the concentration of the invertase.

A method is given for calculating the amounts of fresh dextrose and laevulose that are present at any instant in the solution undergoing inversion. This method is also applicable to the determination of the forms

in which the various hexoses are liberated from glucosides and di- and tri-saccharides by the action of enzymes. It is shown that the dextrose formed from sucrose by the action of invertase has $[\alpha]_D^{20}$ 100—125°, and is therefore in the form of α -dextrose ($[\alpha]_D^{20} = 106^\circ$). E. G.

Action of Cold Aqueous Sodium Hydroxide on Cellulose.

II. WALTHER VIEWEG (*Ber.*, 1908, 41, 3269—3275. Compare Abstr., 1907, i, 893).—From 11% to 24% sodium hydroxide solutions, cellulose takes up an amount of the alkali sufficient to form the compound $C_{12}H_{19}O_{10}Na$, and, if the results are plotted, the curve obtained is discontinuous, the point of discontinuity corresponding with the formation of this compound. The curve up to this point is abnormal, but afterwards it is a horizontal straight line pointing to a chemical reaction. It is also shown that the higher the degree of mercerisation the greater the capacity for absorption of sodium hydroxide, and more is taken up at lower than at higher temperatures. Contrary to the statement of Miller (this vol., i, 78), the addition of sodium chloride to the sodium hydroxide increases the absorption, and the conclusion is drawn that the reaction is physical as well as chemical. W. R.

Walden's Inversion. III. EMIL FISCHER and HELMUTH SCHEIBLER (*Ber.*, 1908, 41, 2891—2902. Compare Abstr., 1907, i, 192; this vol., i, 324).—The optical properties of valine (α -aminoisovaleric acid) are not altered by the double transformation into bromofatty acid and back to amino-compound. There are some reasons for thinking that this is due rather to a double Walden inversion than to its absence.

d-Valine forms *l*- α -bromoisovaleric acid, which, when coupled with valine and treated with ammonia, yields *l*-valyl-*d*-valine. This dipeptide yields racemic valine on hydrolysis, and forms also *trans*-valine anhydride. *l*-Valine, when treated with nitrosyl bromide, forms *d*- α -bromoisovaleric acid, from which *d*-valylglycine is obtained on treatment with ammonia and *d*-valine on subsequent hydrolysis. Thus *d*-bromoisovaleric acid yields *l*-valine, and *d*-bromoisovaleryl-glycine yields *d*-valine. The ester of *d*-bromoisovaleric acid likewise appears to yield *d*-valine.

Both silver oxide and potassium hydroxide, acting on *d*-bromoisovaleric acid, give rise to the same hydroxyisovaleric acid, which is dextrorotatory in alkaline solution. *d*-Bromoisovaleryl-glycine, treated with silver oxide in a similar manner, yields a highly optically active hydroxy-compound, which, on hydrolysis, gives rise to the same dextrorotatory hydroxyisovaleric acid. This acid is also formed from *l*-valine by the action of nitrous acid, which in this case produces inversion, an observation quite contrary to what has been observed for alanine or aspartic acid.

The α -hydroxyisovaleric acid, prepared by means of silver oxide, has in normal sodium hydroxide, $[\alpha]_D^{20} + 11.8^\circ$; prepared using potassium hydroxide, it has $[\alpha]_D^{20} + 12.0^\circ$; nitrous acid yields the same compound, $[\alpha]_D^{20} + 12.2^\circ$.

d-*Bromoisovalerylchloride*, $C_4H_7\cdot CHBr\cdot COCl$, has b. p. $54-55^\circ/13$ mm. (corr.). *d*-*Bromoisovalerylglycine*,

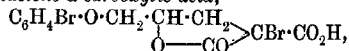
$C_4H_7\cdot CHBr\cdot CO\cdot NH\cdot CH_2\cdot CO_2H$, forms large, well-formed, radiating prisms, m. p. $119-120^\circ$ (corr.) to a colourless liquid after sintering at 115° (corr.). It has $[\alpha]_D^{20} + 47.5^\circ$. The *α*-hydroxyisovaleric acid obtained from this by means of silver oxide has $[\alpha]_D^{20} + 11.8^\circ$.

The zinc salt of active *α*-hydroxyisovalerylglycine crystallises in silvery, four-cornered prisms, $[\alpha]_D^{20} + 48.7^\circ$ in water and $+36.1^\circ$ in *N*-hydrochloric acid.

E. F. A.

Preparation of *α*-Amino- γ - δ -dihydroxy-*n*-valeric Acid. EWU FISCHER and ADOLF KRÄMER (*Ber.*, 1908, 41, 2728-2739).—In view of the general occurrence of serine in the scission products of proteins, and the formation of hydroxyproline in the hydrolysis of gelatin, it is probable that other aliphatic aminohydroxy-acids are formed by the decomposition of proteins. Among these are to be expected derivatives of *n*-valeric and *n*-hexoic acids, owing to their relation to ornithine and lysine. *α*-Amino- δ -hydroxy-*n*-valeric acid has already been synthesised by Sørensen (*Abstr.*, 1905, i, 749), and converted into *r*-proline by hydrochloric acid. The authors have now made experiments on the synthesis of an aminodihydroxy-acid, which should stand in a similar relation to hydroxyproline, and to this end have employed the method used by Fischer and Blumenthal (*Abstr.*, 1907, i, 191) for the synthesis of *α*-amino- γ -hydroxybutyric acid.

γ-Chloro- β -hydroxy-*α*-phenoxypropane, $OPh\cdot CH_2\cdot CH(OH)\cdot CH_2Cl$, prepared by heating epichlorohydrin with phenol under pressure, is a colourless, viscid oil, b. p. $152-153^\circ/12$ mm., with a rancid odour and a bitter taste; when treated with ethyl sodiomalonate and the product hydrolysed, it yields δ -phenoxy- γ -valerolactone-*α*-carboxylic acid, $OPh\cdot CH_2\cdot CH\cdot CH_2\cdot \begin{smallmatrix} O \\ \parallel \\ O-CO \end{smallmatrix} > CH\cdot CO_2H$, in slender needles, sintering at $93-96^\circ$ (corr.) and evolving gas at a higher temperature. A chloroform solution of this gives with bromine, *α*-bromo- δ -bromophenoxy- γ -valerolactone-*α*-carboxylic acid,



forming slender needles, m. p. 157° (corr.), losing carbon dioxide when heated, with the production of *α*-bromo- δ -bromophenoxy- γ -valerolactone, which crystallises in slender, microscopic needles or rods and sometimes prisms, m. p. 128° (corr.). By the action of ammonia, this is converted into *α*-amino- δ -bromophenoxy- γ -valerolactone, which forms microscopic, slender, colourless leaflets, m. p. 230° (corr., decomp.); the hydrochloride has m. p. 229° (corr., decomp.), and the hydrobromide has m. p. 235° (corr., decomp.). The aminolactone dissolves in alkalis, forming salts of *α*-amino- γ -hydroxy- δ -bromophenoxyvaleric acid, of which the silver salt was analysed. When the aminolactone is heated under pressure with concentrated hydrobromic acid and the product hydrolysed (to remove combined

romine), a mixture of amino-acids is obtained, which can be separated by means of the copper salts. The sparingly soluble product is the copper salt of (α)- γ -hydroxyproline (Leuchs, Abstr., 1905, i, 545), and the readily soluble one is probably copper α -amino- γ -dihydroxy-n-valerate; it forms small, dark blue leaflets. The acid,



prepared from the copper salt by treatment with hydrogen sulphide, forms bushy needles, m. p. 160—165°. J. C. C.

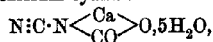
Action of Certain Oxidising Agents on Thiocyanic Acid. Z. BONGIOVANNI (*Gazzetta*, 1908, 38, ii, 299—301).—The author has shown previously (Abstr., 1907, i, 833; this vol., i, 770) that the product of the reaction between a ferric salt and a thiocyanate is a salt of the composition $\text{Fe}(\text{CNS})_3$, and not a peroxygenated salt, as Farugi (Abstr., 1905, i, 176) supposes. Further experimental evidence in support of this view is now adduced.

When freshly-prepared nickel or cobalt hydroxide is treated for some days with a 3% thiocyanic acid solution, it is converted into the nickelous or cobaltous salt of the acid without any formation of red hyper-acid occurring. Further, the addition of acid produces no red coloration, as it should do if the reaction resulted in the formation of a colourless salt of the type $\text{Na}_3\text{C}_3\text{N}_3\text{S}_3\text{O}_3$. It is found that the thiocyanic acid is not decomposed, owing to the strong oxidising action of the nickel or cobalt hydroxide, and that the salt formed has no oxidising properties. Molybdenum thiocyanate has a red colour, but is only formed in presence of a reducing agent, which cannot, of course, determine the formation of a hyper-acid. T. H. P.

Supposed Ammoniacal Fermentation of Cyanamide. CELSO JUPIANI (*Gazzetta*, 1908, 38, ii, 358—417).—The experiments of Löhnis (Abstr., 1905, ii, 412) and of Löhnis and Sabaschnikoff (this vol., ii, 120) on the formation of ammonia from calcium cyanamide by the action of bacteria led to erroneous conclusions, because (1) "Kalk-tickstoff" gradually undergoes change, and only when freshly prepared does it contain one single nitrogenous compound, calcium cyanamide; (2) after sterilisation by heating, solutions of calcium cyanamide contain no trace of the latter, and (3) many amidic substances, besides ammonium salts, yield ammonia when their solutions are distilled with magnesia at the ordinary pressure. From the fact that "Kalk-tickstoff" solutions which have not been sterilised by heating do not undergo bacterial decomposition, Löhnis assumes that the action of heat results in the conversion of the asymmetric form of cyanamide, $\text{N}:\text{C}\cdot\text{NH}_2$, into the symmetrical form, $\text{NH}:\text{C}:\text{NH}$, which alone is attackable by bacteria; this assumption the author shows to be superfluous.

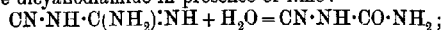
The preparation of pure cyanamide from calcium cyanamide, which is complicated by the tendency of the cyanamide to polymerise into dicyanodiamide, may be affected as follows. One part of "Kalk-tickstoff" is thoroughly mixed with four parts of cold water, and

through the filtered solution, cooled with ice, a current of carbon dioxide is passed. The calcium cyanamidocarboxylate,



thus precipitated may be converted into (1) dicyanodiamide by drying at the ordinary temperature or in an oven, $2(\text{C}_2\text{O}_2\text{N}_2\text{Ca}, 5\text{H}_2\text{O}) = 8\text{H}_2\text{O} + 2\text{CaCO}_3 + (\text{CN}_2\text{H}_2)_2$, or (2) cyanamide by triturating with water and subjecting the mass to the prolonged action of carbon dioxide; $\text{C}_2\text{O}_2\text{N}_2\text{Ca} + \text{H}_2\text{O} = \text{CaCO}_3 + \text{CH}_2\text{N}_2$.

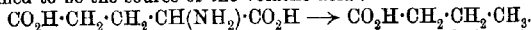
The decomposition of pure cyanamide takes place far more slowly than that of cyanamide accompanied by the secondary products also present in "Kalkstickstoff," and proceeds as well in the presence as in the absence of chloroform, all possibility of bacterial action being thus excluded. The factors which determine the decomposition of cyanamide are calcium, soil, and the secondary products of "Kalkstickstoff," which are capable, in presence of bacteria and of a suitable nutritive medium, of yielding ammonia. These secondary products are found to be: (1) Carbamide, formed by hydrolysis of the cyanamide, (2) Dicyanodiamide, formed by polymerisation. (3) Aminodicyanic acid, $\text{CN}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$, separated in the form of its copper salt, $\text{Cu}(\text{C}_2\text{H}_3\text{ON}_3)_2 \cdot 4\text{H}_2\text{O}$, which, when treated with hydrogen sulphide, gives, not the free acid, but thiobiuret, $\text{CN}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2 + \text{H}_2\text{S} = \text{NH}_2\text{CS}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$. Aminodicyanic acid is formed by the hydrolysis of the dicyanodiamide in presence of lime:



if the hydrolysis is effected by means of acid, dicyanodiamidine is formed: $\text{CN}\cdot\text{NH}\cdot\text{C}(\text{NH}_2)_2\cdot\text{NH} + \text{H}_2\text{O} = \text{NH}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{C}(\text{NH}_2)_2\cdot\text{NH}$. (4) Ammeline, formed by the hydrolysis of melamine, $\text{C}_3\text{H}_6\text{N}_6 + \text{H}_2\text{O} = \text{C}_3\text{H}_3\text{ON}_3 + \text{NH}_3$, itself produced by polymerisation of the cyanamide. When free from acids and bases, a solution of cyanamide remains unchanged at the ordinary temperature.

A list of papers previously published on this subject is given, and the results given therein discussed. T. H. P.

The Biochemical Conversion of Glutamic into *n*-Butyric Acid. WALTHER BRASCH and CARL NEUBERG (*Biochem. Zeitsch.*, 1908, 13, 299—304).—*n*-Butyric acid was shown by Neuberg and Rosenberg to account for about one-third of the volatile fatty acids obtained from the putrefaction of caseinogen. As aminobutyric acid has not been definitely proved to be a product of hydrolysis, glutamic acid was assumed to be the source of the volatile acid:



By actual putrefaction experiments with glutamic acid, butyric acid was obtained to the amount of about 58.6% of the theoretically possible quantity. The other acids formed were formic and succinic acids. Glutaric acid was not obtained, nor did the latter acid yield *n*-butyric acid when treated with putrefying organisms. S. B. S.

α -Camphoramic Acids. MLE. G. FREYRON (*Ann. Chim. Phys.*, 1908, [viii], 15, 278—288).—The author has shown (this vol., i, 827) that α -aminoethylbenzene condenses with camphoric anhydride to form

two ethylbenzene- α -camphoramic acids, which are enantiomorphously related to one another; in the present paper, the substituted α -camphoramic acids obtained similarly from diethylamine, β -aminobutane, and γ -amino-octane are described, but, although each of the last two bases contains an asymmetric carbon atom, they do not furnish stereoisomeric α -camphoramic acids.

Diethyl- α -camphoramic acid, $\text{CO}_2\text{H}\cdot\text{C}_8\text{H}_{14}\cdot\text{CO}\cdot\text{NEt}_2$, crystallises in needles. m. p. 169—170°, and has $[\alpha]_D^{20} + 19.29^\circ$ in alcoholic solution.

β -Butyl- α -camphoramic acid, $\text{CO}_2\text{H}\cdot\text{C}_8\text{H}_{14}\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_4\text{H}_9$, has m. p. 206—208°, and $[\alpha]_D^{20} + 28.80^\circ$ in alcoholic solution.

δ -Amino-octane, $\text{C}_8\text{H}_{17}\cdot\text{CH}(\text{NH}_2)\cdot\text{C}_4\text{H}_9$, prepared by reducing the oxime, $\text{C}_8\text{H}_{17}\cdot\text{C}(\text{NOH})\cdot\text{C}_4\text{H}_9$, b. p. 116—117°/20 mm., by means of sodium and alcohol, has b. p. 64—65°/18 mm., and forms a *hydrochloride*, m. p. 194—195°, and a *carbonate*; the corresponding *carbamide*,

$\text{NH}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_8\text{H}_{17}$, has m. p. 166—168°. When heated for six hours at 120° with camphoric anhydride, it yields *δ -octyl- α -camphoramic acid*,

$\text{CO}_2\text{H}\cdot\text{C}_8\text{H}_{14}\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_8\text{H}_{17}$, which has m. p. 177—178°, and $[\alpha]_D^{20} + 24.45^\circ$.

M. A. W.

Carbamido-acids. II. and III. FRITZ LIEPICH (*Ber.*, 1908, 41, 2953—2974, 2974—2983. Compare Abstr., 1906, i, 813).—II.—The author finds that the Baumann and Hoppe-Seyler reaction for the preparation of hydantoic acid (this Journ., 1874, 466) is a general reaction of amino-acids, and presents a simple method for the preparation of carbamido-acids. The present communication deals with the α -amino-acids of the glycine series, aspartic and glutamic acids, taurine, and tyrosine. The reaction takes place by heating the amino-acid for six to ten hours with two to three times its weight of carbamide and 200—500 c.c. of baryta water, and is represented by the equation: $\text{NH}_2\cdot\text{R}\cdot\text{CO}_2\text{H} + \text{CO}(\text{NH}_2)_2 = \text{CO}_2\text{H}\cdot\text{R}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2 + \text{NH}_3$.

The carbamido-acids so obtained give flocculent precipitates with mercuric nitrate in the absence of chlorides or free acid, crystalline compounds with alcoholic mercuric chloride or silver nitrate, do not

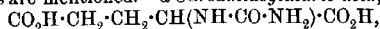
respond to the biuret test, yield hydantoins, $\text{R}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$, by treatment with cold concentrated or with warm dilute mineral acids, and are hydrolysed by hot alkalis or hot concentrated hydrochloric acid, regenerating the amino-acid.

Hydantoic acid (carbamidoacetic acid) has m. p. 163° in a sealed capillary tube (compare Weidel and Roitner, Abstr., 1906, i, 470); the *barium*, *copper*, *lead*, *mercury*, and *silver* salts are described. α -Carbamidopropionic acid and α -carbamidoisobutyric acid are known in the literature as lacturamic acid (Urech, this Journ., 1873, 380) and acetyluramic acid respectively. *α -Carbamidoisovaleric acid*,

$\text{CHMe}_2\cdot\text{CH}(\text{NH}\cdot\text{CO}\cdot\text{NH}_2)\cdot\text{CO}_2\text{H}$, m. p. 176°, separates from water in tufts of long needles; the *barium*, *mercury*, and *silver* salts are described. *α -Carbamidoisohexoic acid* (isobutylhydantoic acid: compare Finner and Spilker, Abstr., 1889, 704) yields crystalline *barium*, *lead*, *copper*, *mercury*, and *silver* salts. The

acids prepared from leucine, obtained from different sources, show slight differences in solubility, m. p., and per cent. of nitrogen; these are explained by specific differences in the original leucines.

α-Carbamidosuccinic acid, $\text{CO}_2\text{H}\cdot\text{CH}_2\cdot\text{CH}(\text{NH}\cdot\text{CO}\cdot\text{NH}_2)\cdot\text{CO}_2\text{H}$, m. p. 162°, prepared from aspartic acid, separates from dilute alcohol in rhombic plates or prisms; the *copper, lead, mercury, silver*, and *silver hydrogen* salts are mentioned. *α*-Carbamidoglutamic acid,



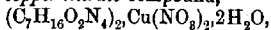
m. p. 150°, crystallises in needles. Taurocarbamie acid, obtained by Salkowsky (this Journ., 1874, 148), and later by Gabriel, is prepared very readily by the author's method. *α*-Carbamido-*β*-*p*-hydroxyphenylpropionic acid, $\text{OH}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{CH}(\text{NH}\cdot\text{CO}\cdot\text{NH}_2)\cdot\text{CO}_2\text{H}$, m. p. 218° (decomp.), is identical with Jaffe's tyrosinehydantoic acid (*Zeitsch. physiol. Chem.*, 1882, 7, 306). The anhydride, tyrosinehydantoin, has m. p. 242–245° (decomp.).

III.—The Baumann and Hoppe-Seyler reaction has been successfully applied to anthranilic, metanilic, sulphanilic, and *β*-aminobutyric acids and to *β*-phenylalanine.

Other general methods for the preparation of carbamido-acids are: (a) an amino-acid and a urethane are boiled with aqueous barium hydroxide. Applied to leucine and *isoamyl* carbamate, a 40% yield of *α*-carbamidoisohexoic acid is obtained. (b) An amino-acid is boiled with an excess of a concentrated aqueous solution of carbamide. The reaction really occurs between the amino-acid and ammonium cyanate; the continuous removal of the latter destroys the equilibrium of the system carbamide \rightleftharpoons ammonium cyanate (compare Walker and Hamby, *Trans.*, 1895, 67, 746). The method is advantageously used in the case of optically active amino-acids, since the reaction occurs in the absence of acid or alkali. Applied to leucine from different sources, the method yields *α*-carbamido-*α*-isohexoic acids, which again show slight differences in physical properties. Similarly, glycine gives ammonium hydantoate, $\text{C}_3\text{H}_5\text{O}_3\text{N}_2(\text{NH}_4)\cdot\text{H}_2\text{O}$; aspartic acid is converted into *α*-carbamidosuccinic acid, which is isolated as the *hydantoin*, $\text{C}_5\text{H}_8\text{O}_4\text{N}_2$, m. p. 208° (decomp.), whilst tyrosine very readily yields the ammonium salt of the corresponding carbamido-acid. Leucine and potassium cyanate in boiling aqueous solution give an 83% yield of the carbamido-acid. (c) Prolonged boiling of an aqueous solution of an amino-acid and guanidine carbonate. The method has been used successfully with leucine and with tyrosine. C. S.

Synthesis of *α*-ω-Aminoguanidinehexoic Acid. FRITZ HECKEL (*Monatsh.*, 1908, 29, 779–785).—Lysine, which was the starting point of this synthesis, was prepared from the sparingly soluble phosphotungstates obtained by fractional precipitation with phosphotungstic acid of the acid-hydrolytic products of casein; the precipitate is dissolved in aqueous ammonia, treated with barium hydroxide, and the mixture of amino-acids separated by means of the picrates, that of arginine being more soluble than lysine picrate. The latter is then converted into the dihydrochloride. This is treated in aqueous solution with silver cyanamide; after separating unchanged

lysine by means of silver nitrate, an uncrystallisable syrup of the base was obtained. The copper nitrate compound,



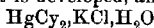
forms dark blue, crystalline platelets; the anhydrous salt has m. p. 210° (decomp.). The compound of the nitrate with silver nitrate, $C_7H_{16}O_2N_4 \cdot AgNO_3 \cdot HNO_3$, forms white needles, blackening at 60° , m. p. 125° (decomp.). The nitrate has $[\alpha]_D + 5.37^\circ$. J. C. C.

Reduction of Nitriles in Neutral Solutions. HEINRICH BRUNNER and A. RAPIN (*Chem. Zentr.*, 1908, ii, 676—677; from *Schweiz. Woch. Chem. Pharm.*, 1908, 46, 455—457).—Reduction is effected most rapidly by using a magnesium-copper couple, the reaction only taking place with Devarda's alloy when warmed. In both cases the action is identical, but the yield is better when Devarda's alloy is used. Hydrogen cyanide gives, on reduction, formaldehyde and ammonia. Acetonitrile gives acetaldehyde, ethylamine, and ammonia. Propionitrile gives propaldehyde, ethylamine, methylethylamine, methylpropylamine, and ammonia. *n*-Butyronitrile yields butaldehyde, butylamine, dibutylamine, and ammonia. Benzonitrile gives benzaldehyde, benzylamine, dibenzylamine, diphenylethylenimide, $\begin{matrix} CHPh \\ CHPh \end{matrix} > NH$, and ammonia. *o*-Toluenitrile gives *o*-xylamine. *m*-Toluenitrile gives *m*-tolualdehyde, *m*-xylamine, *m*-dixylamine, and ammonia. *p*-Toluenitrile yields *p*-tolualdehyde, *p*-xylamine, *p*-dixylamine, ditolyethylenimide, $\begin{matrix} CHMePh \\ CHMePh \end{matrix} > NH$, and ammonia. *o*-Cyanodiphenylmethane gives *o*-aminodiphenylmethane, $CH_2Ph \cdot C_6H_4 \cdot CH_2 \cdot NH_2$. *m*- and *p*-Cyanodiphenylmethane give *m*- and *p*-aminodiphenylmethanes. Diphenylethylenimide forms a very sparingly soluble nitrite and nitrate; the hydrochloride is more soluble, and the sulphate very soluble in water.

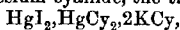
Salts of ditolyethylenimide are more readily soluble.

Catalytic Action of Ethylamine.—It is mentioned that a few drops of this substance hastens the reaction between methyl iodide or ethyl iodide and sodium; the behaviour of acetonitrile in this respect (*Abstr.*, 1901, i, 457) is probably due to ethylamine formed by its reduction. J. V. E.

Preparation of Mercuric Cyanide. ERWIN RUPP and S. GOY (*Chem. Zentr.*, 1908, ii, 773; from *Apoth. Zeit.*, 1908, 23, 373—374).—When mercuric chloride and potassium cyanide are brought together in water, considerable heat is developed, and the compound



crystallises on cooling. The same product is obtained from mercuric cyanide and potassium chloride. In a similar manner, the analogous compound is prepared from mercuric cyanide and potassium bromide, but with mercuric cyanide and potassium iodide, as also from mercuric iodide and potassium cyanide, the triple salt,



crystallises in large needles. The preparation of pure mercuric cyanide is effected by treating sodium cyanide with mercuric sulphate

in the presence of water; the solution becomes very warm, and, on cooling, practically pure mercuric cyanide separates out. J. V. E.

Saturated Compounds of the cycloHexane Group. NICOLAI D. ZELINSKY (*Ber.*, 1908, 41, 2676—2680).—cycloHexylpropionic acid is prepared by the following series of reactions. ω -Iodomethylcyclohexane (b. p. 82—83°/10 mm., D_4^{20} 1.5310) is condensed with ethyl sodiomalonate, and the ethyl hexahydrobenzylmalonate formed, b. p. 145—155°/12 mm., hydrolysed to hexahydrobenzylmalonic acid, m. p. 106.5°. On heating the latter at 170°, it loses carbon dioxide and yields cyclohexylpropionic acid, b. p. 143.5°/11 mm., D_4^{20} 0.9966, n_D^{20} 1.4364; the amide forms thin, soft needles, m. p. 120° (corr.).

[With D. SCHWEDOFF.]—cycloHexylglycollic acid, prepared by treating the hydrogen sulphite compound of hexahydrobenzaldehyde with potassium cyanide and hydrolysis of the resulting nitrile, crystallises from acetone in silvery scales, m. p. 166°. ω -Nitromethylcyclohexane, prepared by the interaction of ω -iodomethylcyclohexane and silver nitrite, has b. p. 98°/10 mm., D_4^{20} 1.0473, n_D^{20} 1.4688. From its salts, acids precipitate the labile act-form as a heavy, golden-yellow oil, which slowly resumes the stable form.

2-Chloro-1-methylcyclohexane, prepared by the action of fuming hydrochloric acid on 1-methylcyclohexan-2-ol, is stereoisomeric with the compound obtained by the action of phosphorus pentachloride. It has b. p. 91—92°/100 mm., D_4^{20} 0.9699, n_D^{20} 1.4575, and with magnesium and carbon dioxide furnishes *cis*-1-methylcyclohexane-2-carboxylic acid, b. p. 122—123°/10 mm., of which the amide forms slender needles, m. p. 151—153°. [The author has overlooked the fact that this *cis*-acid has already been described by Freer and Perkin, jun. (*Trans.*, 1888, 53, 208), and Goodwin and Perkin, jun. (*Trans.*, 1895, 67, 125).]

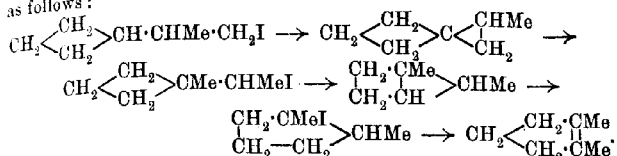
J. C. C.

Isomeric Changes in the Transformations of cycloButyl-dimethylcarbinol. NICOLAI M. KIJNER (*J. Russ. Phys. Chem. Soc.*, 1908, 40, 994—1015. Compare this vol., i, 530).—The transformations of cyclobutyl-dimethylcarbinol as yet studied are all abnormal changes, and the preparation from this carbinol of a hydrocarbon of the tetramethylene series has not yet been effected, all the methods employed for this purpose yielding products of isomeric change. This isomeric change of the carbon skeleton of cyclobutyl-dimethylcarbinol either leads exclusively to the formation of cyclopentane derivatives or is complicated by the formation of cyclohexane derivatives, according to the conditions under which it takes place.

The formation of derivatives of 1:1-dimethylcyclopentane from cyclobutyl-dimethylcarbinol by the action of hydriodic (or hydrobromic) acid

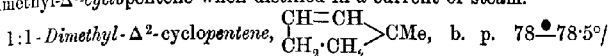
is probably effected by way of the stages: $\text{CH}_2 \begin{smallmatrix} \text{CH}_2 \\ \text{CH}_2 \end{smallmatrix} \text{CH} \cdot \text{CMe}_2\text{I} \rightarrow \begin{smallmatrix} \text{CH}_2 \cdot \text{CH} \\ \text{CH}_2 \cdot \text{CH} \end{smallmatrix} \text{CMe}_2 \rightarrow \begin{smallmatrix} \text{CH}_2 \cdot \text{CHI} \\ \text{CH}_2 \cdot \text{CH}_2 \end{smallmatrix} \text{CMe}_2$; both the bromide and iodide formed, when treated with alcoholic potassium hydroxide, yield 1:1-dimethyl- Δ^2 -cyclopentene, the structure of which is demonstrated

by the formation of *aa*-dimethylglutaric acid on oxidation. The formation of 1:2-dimethyl- Δ^1 -cyclopentene by the action of hydriodic or hydrobromic acid on cyclobutyldimethylcarbinol probably proceeds as follows:



The structure of this hydrocarbon, which is regarded as identical with Rénard's heptene and with that obtained by Maquenna from perseitol (see this vol., i, 530), is deduced from the fact that it yields γ -acetobutyric acid on oxidation.

The action of hydrobromic acid on cyclobutyldimethylcarbinol yields: (1) 2-bromo-1:1-dimethylcyclopentane, $\text{C}_7\text{H}_{13}\text{Br}$, b. p. 167°/764 mm., D_4^{20} 1.2523, n_D^{20} 1.4797, which gives 1:1-dimethyl- Δ^2 -cyclopentene (*vide infra*) on treatment with alcoholic potassium hydroxide; (2) an unstable bromo-derivative, which yields 1:2-dimethyl- Δ^1 -cyclopentene when distilled in a current of steam.



754 mm., D_4^{20} 0.7580, n_D^{20} 1.4190, has an odour resembling that of naphthalene, gives an intense green coloration when it is heated with concentrated sulphuric acid and alcohol added to the mixture, and yields *aa*-dimethylglutaric acid when oxidised with nitric acid.

The action of hydriodic acid on cyclobutyldimethylcarbinol also yields two products: (1) 2-iodo-1:1-dimethylcyclopentane, $\text{C}_7\text{H}_{13}\text{I}$, b. p. 98–99°/40 mm., D_4^{20} 1.5020, n_D^{20} 1.5240, which gives 1:1-dimethyl- Δ^2 -cyclopentene when treated with alcoholic potassium hydroxide; (2) an unstable iodo-derivative, which gives 1:2-dimethyl- Δ^1 -cyclopentene when distilled in a current of steam.

The reduction of 1:1-dimethyl- Δ^2 -cyclopentene by Sabatier's method, and the reduction of 2-iodo-1:1-dimethylcyclopentane by means of a zinc-copper couple, both yield 1:1-dimethylcyclopentane (compare Abstr., 1905, i, 772).

The reduction of 1:1-dimethyl- Δ^2 -cyclopentene by means of sulphuric acid yields 1:2-dimethylcyclopentane, which is also obtained, together with methylcyclohexane, when the reduction is effected by means of hydriodic acid. The reduction of 2-bromo-1:1-dimethylcyclopentane by hydriodic acid also yields a mixture of 1:2-dimethylcyclopentane and methylcyclohexane.

The oxidation of 1:2-dimethyl- Δ^1 -cyclopentene by means of nitric acid gives, as main products, acids such as oxalic and succinic, which are accompanied by a small proportion of a compound, $\text{C}_7\text{H}_{13}\text{O}_4\text{N}$, m. p. 202° (decomp.), the nature of which was not determined.

1:2-Dimethylcyclopentane, $\begin{array}{c} \text{CH}_2 \cdot \text{CHMe} \\ \text{CH}_2 \cdot \text{CH}_2 \end{array} \text{CHMe}$, prepared by the reduction of 1:2-dimethyl- Δ^2 -cyclopentene by Sabatier's method, is a liquid, b. p. 92.7–93°/762 mm., D_4^{20} 0.7534, n_D^{20} 1.4126, which is

completely dissolved when heated with nitric acid (D 1.52), giving products which are under investigation.

T. H. P.

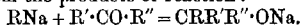
Electro-Syntheses. III. SIMA M. LOSANITSCH (*Ber.*, 1908, 41, 2683—2688. Compare this vol., ii, 32).—In the apparatus previously described, benzene vapour alone, or mixed with other gases, is submitted to the silent electric discharge. Benzene alone gives a solid, insoluble in benzene; a liquid, soluble in ether or benzene; a solid, soluble in benzene, insoluble in ether. All three have the formula $C_{24}H_{25}$, and absorb oxygen. In presence of air, the resulting product is a dark, viscous substance containing nitrogen; with hydrogen, a yellow oil, $C_{22}H_{34}$, is obtained; with methane, a viscous, yellowish-red liquid, $C_{28}H_{36}$; with ethylene, a viscous, brownish-red liquid, $C_{28}H_{34}$; with acetylene, a yellowish-brown mass containing two substances, one soluble, the other insoluble, in benzene. Both have the composition $C_{48}H_{49}$, and absorb oxygen. The product with carbon monoxide is a dark viscous liquid, containing one substance soluble in ether, and a second, insoluble, which absorbs oxygen, yielding substances of the composition $C_{22}H_{29}O_2$ and $C_{20}H_{20}O_3$ respectively, and with carbon disulphide a dark solid, $C_8H_{10} \cdot 6CS_2$.

The behaviour of ammonia, mixed with other gases, under the influence of the silent discharge has been examined. With an equal volume of ethylene, it yields a yellow basic oil, $C_{10}H_{21}N_3$, which absorbs oxygen; with acetylene, a dark red basic liquid, $C_{18}H_{30}N_4$, is formed, which absorbs oxygen; with benzene, a reddish-brown oily liquid, $C_{16}H_{24}N_2$, is formed, which absorbs oxygen.

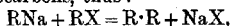
In his answer to Loeb's criticisms (this vol., i, 117), the author states that the analyses of the preceding substances give concordant results only after the preparations have been saturated with oxygen by exposure to the air for about a month.

C. S.

Syntheses by means of Sodium and Alkyl Halides. PAUL SCHORIGIN (*Ber.*, 1908, 41, 2711—2717).—In continuation of his former work (*Abstr.*, 1907, i, 753), the author has investigated the action of sodium and alkyl halides on ketones. Here, as before, it is probable that the sodium alkyl is formed as an intermediate product, for the corresponding tertiary alcohols (or their dehydration products) can be isolated from the products of reaction:



In addition to this, several other reactions take place, for example, the formation of hydrocarbons, thus:



and the formation of compounds of sodium with the ketones. The production of compounds of high b. p. also occurs, so that the yield of tertiary alcohols is low, varying from 5% to 10%.

The nature of the blue substances which are formed in the Fittig and Wurtz syntheses is discussed, and the author suggests that they may consist of a blue modification of sodium iodide or possibly salts of sodium quadratoxide.

aa-Diphenyl- δ -methyl- Δ^a -amylene, $CPh_2 \cdot CH \cdot CH_2 \cdot CHMe_2$, prepared by the action of sodium on a mixture of benzophenone and isomyl

bromide in ethereal solution, is a colourless liquid, b. p. $178^{\circ}/16$ mm., D_4^{20} 0.9907, D_4^{25} 0.9725, n_D^{20} 1.57463. It is not formed by the action of magnesium *isoamyl* bromide on benzophenone. When ethyl iodide is substituted for *isoamyl* iodide in the above reaction, diphenylethylcarbinol is produced. The interaction of sodium, acetophenone, and *isoamyl* iodide leads to the formation of *phenylmethylisoamylcarbinol*, a colourless liquid, b. p. $135-137^{\circ}/15$ mm., D_4^{20} 0.9608, D_4^{25} 0.9450, n_D^{20} 1.50596.

When a stream of carbon dioxide is led through a mixture of sodium, benzene, and *isoamyl* bromide, a blue substance (referred to above) is produced, which is indifferent towards carbon dioxide, and, in addition, a small amount of benzoic acid is formed.

By the action of carbon dioxide on a mixture of sodium and *isobutyl* bromide in ethereal solution, a very small quantity of *isovaleric* acid is obtained. J. C. C.

Reduction of Nitro-compounds with Zinc Dust and Acetic Acid. GUSTAV HELLER (*Ber.*, 1908, 41, 2689—2692).—The discovery of a new step in the reduction of *o*-nitromandelonitrile by zinc dust and 33% acetic acid at a low temperature, whereby a double compound of a dihydroxylamino- and a hydroxylamino-derivative is produced (*Abstr.*, 1906, i, 585; Heller and Sourlis, this vol., i, 208), has led the author to submit a number of other *o*-nitro-compounds to this process of reduction. It is remarkable that the presence of an ortho-substituent appears to favour the reduction, for, whilst *o*-nitromandelonitrile is readily reduced at 0° , nitrobenzene, even at the ordinary temperature, is scarcely attacked, yielding only a small amount of phenylhydroxylamine.

o-Nitrobenzaldehyde gives a solution containing *o*-aminobenzaldehyde and anthranil.

[With JULIUS SÖLLING.]—*o*-Nitrobenzoic acid, when reduced with zinc dust and 50% acetic acid in presence of sodium acetate at $35-40^{\circ}$, yields a mixture of benzidine-2:2'-dicarboxylic acid and azoxybenzoic acid; the latter is also produced when the reduction is carried on in ammoniacal solution. J. C. C.

Products of the Nitration of *o*-Chloronitrobenzene. IWAN OSTROMISLENSKY (*J. pr. Chem.*, 1908, [ii], 78, 260—262).—According to Jungfleisch (*Ann. Chim. Phys.*, 1868, [iv], 15, 239), 1-chloro-2:4-dinitrobenzene, formed by nitration of chlorobenzene or of *o*-chloronitrobenzene, is obtained in a labile modification, m. p. 43° , and a stable modification, m. p. 50° . As the reduction products of these two modifications were found not to be identical, Beilstein considered (*Handbuch*, 3rd ed., II, 84) the labile form to be 1-chloro-2:6-dinitrobenzene, and states that this undergoes intramolecular change into the 1:2:4-isomeride when brought into contact with a crystal of the latter. As no other such transformation of a benzene derivative is known, the present author undertook a fresh study of the nitration of *o*-chloronitrobenzene, and has found that, whilst the main product is 1-chloro-2:4-dinitrobenzene, there is formed also 1-chloro-

2:6-dinitrobenzene, $C_6H_3Cl(NO_2)_2$, which crystallises in needles, m. p. 38° , yields 2:4-dinitrophenol on treatment with warm concentrated potassium hydroxide, and is not identical with Jungfleisch's labile modification.

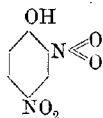
G. Y.

Structure of Benzene. I. Polymorphic Modifications of Aromatic Compounds. II. Homogeneity of the ortho- and of the meta-Derivatives of Benzene. III. The Two Chemically Isomeric 2:4-Dinitrophenols. IWAN OSTROMISLENSKY (*J. pr. Chem.*, 1908, [ii], 78, 263—279. Compare Abstr., 1907, i, 120, 596; preceding abstract).—It is found that both modifications of 1-chloro-2:4-dinitrobenzene, (1) on reduction with tin and hydrochloric acid or with stannous chloride (Claus and Stiebel, Abstr., 1887, 810), yield two chloronitroanilines, m. p. 117.8° and $101-102^\circ$; (2) when treated with aniline, form 2:4:2':4'-tetranitrodiphenylamine, m. p. $155-156^\circ$, and (3) on treatment with cold concentrated potassium hydroxide, are converted into 2:4-dinitrophenol, m. p. 113.2° . The two modifications are, therefore, chemically identical. Attempts to prepare two modifications of either of the chloronitroanilines were unsuccessful. It is suggested that Jungfleisch's chloronitroaniline, m. p. 89° , may be 2-chloro-3-nitroaniline, derived from 1-chloro-2:6-dinitrobenzene.

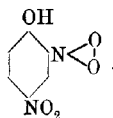
The existence of the liquid modification of 1-chloro-3:4-dinitrobenzene (Laubenheimer, this Journ., 1876, ii, 294) is confirmed. A mixture of the liquid and solid modifications is obtained when the solid modification is recrystallised from ether. The action of ammonia on the ethereal solution of this mixture leads to the formation of a *chloronitroaniline*, $C_6H_5O_2N_2Cl$, which crystallises in yellow prisms, melts when handled, has a blue fluorescence, is not identical with either of the known 3-chloro-6-nitro- and 4-chloro-6-nitro-anilines, and is probably a derivative of the liquid modification of 1-chloro-3:4-dinitrobenzene. It is considered that these cases of isomerism may depend on a difference in the structure of a nitro-group.

With the object of proving the homogeneity of the ortho- and of the meta-derivatives of benzene, *o*-chloronitro-, *m*-chloronitro-, *o*-dinitro-, 1-chloro-3:4-dinitro-, and 1-chloro-2:4-dinitro-benzene were subjected to fractional separation in the manner employed by Groos for the separation of the rare earths. The number of fractions obtained varied between 60 and 125. All the fractions of each substance were identical as to the m. p. and the crystalline constants.

The two modifications of 2:4-dinitrophenol are discussed, and it is suggested that here, also, the isomerism arises from a difference in the structure of a nitro-group, the two modifications having the constitutions:



and



G. Y.

Transformation of Phenylnitromethane [ω -Nitrotoluene] into Phenyldinitromethane [Di- ω -nitrotoluene]. GIACOMO PONZIO (*Gazzetta*, 1908, **38**, ii, 417—420).—*Phenyldibromonitromethane* [Di- ω -bromo- ω -nitrotoluene], $\text{CPhBr}_2\cdot\text{NO}_2$, prepared by the action of an alkaline solution of bromine on the sodium derivative of phenylnitromethane, is a colourless oil heavier than water, and has a pungent odour. On treatment with potassium hydroxide and potassium nitrite in alcoholic solution, it is converted into the potassium derivative of phenyldinitromethane; the reaction is possibly expressed by the equation :

$$\text{CPhBr}_2\cdot\text{NO}_2 + \text{KNO}_2 + 2\text{KOH} = \text{CPhK}(\text{NO}_2)_2 + 2\text{KBr} + \text{H}_2\text{O} + \text{O},$$

the oxygen oxidising part of the alcohol (compare ter Meer, *Abstr.*, 1876, **30**, 185). Phenyldinitromethane is obtained from its potassium derivative by the action of dilute sulphuric acid. T. H. P.

Partial Electrolytic Reaction of Polynitro-compounds in the Presence of Vanadium Compounds. HANS HOFER and FRITZ JAKOB (*Ber.*, 1908, **41**, 3187—3199).—The use in electrolytic reactions of inorganic compounds which are themselves reduced at the cathode, modifies the reduction of nitro-compounds. Vanadium, occurring as it does in so many forms of oxidation, gives a potential scale extending from the low oxidation potential of vanadic acid to the high reduction potential of bivalent vanadium salts. It should therefore be suitable for the partial reduction of polynitro-compounds, as it gives the necessary high potential (compare Brand and Zöller, *Abstr.*, 1906, i, 80; 1907, i, 755).

The preliminary work consisted in measuring the potential of the various vanadium compounds, with the result: vanadic acid -1.17 , vanadyl sulphate -0.76 , vanadic sulphate -0.23 volt (e_h for mercurousulphate electrode -0.69 volt). From the Peters-Nernst formula, the "equilibrium potential" for mixtures of salts can be calculated, and the reduction tension determined.

The cathode was contained in a narrow beaker containing the vanadyl sulphate, sulphuric acid, alcohol, and the polynitro-compound; the lead anode was placed in a porous cell containing normal sulphuric acid at $50-60^\circ$.

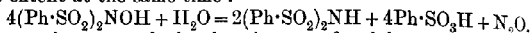
2:4-Dinitrotoluene, with a platinum cathode and potential 0.03 volt, gave chiefly *p*-nitrotoluidine with tolylenediamine and 2:2'-dinitro-4:4'-azoxytoluene. With a potential of $0.10-0.15$ volt, more of the azoxy-compound was produced. Lead and zinc are very much less suitable as cathodes; with copper, no azoxy-derivative was obtained, but some dinitrotoluene was recovered unchanged.

Platinum was used as electrode in the remaining experiments. 2:6-Dinitrotoluene gave 2:2'-dinitro-6:6'-azoxytoluene, 2-nitro-6-toluidine, and a little of the corresponding diamine. With 2:4:6-trinitrotoluene, only 2:6-dinitro-4-aminotoluene was obtained. It sinters at 108° , m. p. 127° , to a viscous liquid, which becomes mobile at 150° (Beilstein gives m. p. 168° ; *Abstr.*, 1880, 636); its constitution was determined by preparing 2:6-dinitrotoluene by digesting the diazo-compound with alcohol.

m-Dinitrobenzene gave *m*-nitroaniline, *m*-phenylenediamine, and the

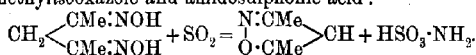
dinitroazoxybenzene; 2:4-dinitrophenol yielded *o*-nitro-*p*-amino-phenol, and picric acid, picramic acid.
W. R.

Simple Method of Preparing the Imides of the Aromatic Sulphonic Acids. TAMEMASA HAGA (*J. Coll. Sci. Tokyo*, 1908, 25, xiii, 1—30).—Very little work has been done hitherto on the aromatic sulphonimides (compare Farbwerke vorm. Meister, Lucius & Brünig, Abstr., 1902, i, 364). It is found that these compounds can be readily obtained by reducing aromatic sulphonylhydroxylamines, preferably with sulphur dioxide, thus: $(\text{Ph}\cdot\text{SO}_2)_2\text{NOH} + \text{SO}_2 + \text{H}_2\text{O} = (\text{Ph}\cdot\text{SO}_2)_2\text{NH} + \text{H}_2\text{SO}_4$. The following reaction also takes place to some extent at the same time:



This reaction proceeds in the absence of sulphur dioxide, for it is found that dibenzenesulphonylhydroxylamine in aqueous alcoholic solutions decomposes mainly according to the last equation, a small quantity of tribenzenesulphonylhydroxylamine also being formed. It is probable that the sulphonate, $(\text{Ph}\cdot\text{SO}_2)_2\text{N}\cdot\text{SO}_3\text{H}$, is formed as an intermediate product during the reduction of dibenzenesulphonylhydroxylamine with sulphur dioxide, but its existence could not be established. Dibenzenesulphonylhydroxylamine, when reduced with sodium amalgam, yields a quantity of benzenesulphonic acid, with liberation of ammonia; the yield of dibenzenesulphonimide is very small.

Krafft and Bourgois (Abstr., 1892, 700) have shown that an oximino-nitrogen atom attached to carbon alone becomes sulphonated when treated with sulphur dioxide, and is at the same time detached from the carbon. When, however, a compound containing two oximino-radicles united to two separate carbon atoms in the molecule is reduced with sulphur dioxide, only one of the nitrogen atoms is separated in the form of amidosulphonic acid; the other nitrogen atom remains united to carbon. For example, when acetylacetonedioxime in aqueous solution is treated with sulphur dioxide, it is converted into 3:5-dimethylisooxazole and amidosulphonic acid:

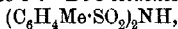


The non-removal of one of the nitrogen atoms is not due to the tendency of forming a ring compound, since acetylacetonedioxime, when similarly treated, yields amidosulphonic acid and an oily substance, which still contains a nitrogen atom combined with carbon in a form capable of yielding hydroxylamine on hydrolysis.

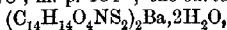
Diphenylsulphonylhydroxylamine (dibenzenesulphohydroxamic acid) crystallises with 1Et·OH and sometimes with 2H₂O; the anhydrous substance has m. p. 132°; Angeli, Angelico, and Scurti give m. p. 126° (Abstr., 1902 i, 765). *Dibenzenesulphonimide*, $(\text{Ph}\cdot\text{SO}_2)_2\text{NH}$, probably crystallises with 1½H₂O; it forms thick prisms, and when anhydrous has m. p. 157°; the barium, $\text{C}_{24}\text{H}_{20}\text{O}_8\text{S}_4\text{N}_2\text{Ba}\cdot 2\text{H}_2\text{O}$, mercuric, $\text{C}_{24}\text{H}_{20}\text{O}_8\text{S}_4\text{N}_2\text{Hg}$, potassium, $\text{C}_{12}\text{H}_{10}\text{O}_4\text{S}_2\text{NK}$, mercurous, silver, sodium, and ammonium salts are described.

Di-o-tolylsulphonylhydroxylamine, $(\text{C}_6\text{H}_4\text{Me}\cdot\text{SO}_2)_2\text{NOH}$, prepared by

the action of nitrous acid on *o*-toluenesulphonic acid, crystallises with $1\frac{1}{2}H_2O$ in small prisms, m. p. $106-107^\circ$; it is converted by an excess of nitrous fumes evolved from arsenious oxide and nitric acid ($D 1.32$) into *tri-o-tolylsulphonylhydroxylamine*, $(C_6H_4Me\cdot SO_2)_3NO$, crystallising in small needles, m. p. 129.5° . *Di-o-toluenesulphonimide*,



crystallises with H_2O in thick plates or short prisms; the anhydrous substance sinters at 178° , m. p. 184° ; the *barium* salt,



forms minute needles.

Di-p-toluenesulphonimide, $(C_6H_4Me\cdot SO_2)_2NH$, forms crystals, m. p. $168-170^\circ$. W. H. G.

Oximes and Imides of Benzenedisulphonic Acids. TSUNEO SZUKI (*J. Coll. Sci. Tokyo*, 1908, **25**, xiv, 1-21).—Mol.-wt. determinations show that the substance described by Autenrieth and Hennings as *m*-benzenedisulphonylhydroxamic acid, $C_6H_4\cdot(SO_2)_2\cdot NOH$ (compare Abstr., 1902, i, 389), is bimolecular, and probably has the formula $OH\cdot N < \begin{smallmatrix} SO_2\cdot C_6H_4\cdot SO_2 \\ SO_2\cdot C_6H_4\cdot SO_2 \end{smallmatrix} > N\cdot OH$. It crystallises with alcohol, $[C_6H_4(SO_2)_2NOH]_2$, $1\frac{1}{2}EtOH$, and with acetone, $[C_6H_4(SO_2)_2NOH]_2\cdot \frac{2}{3}COMe_2$.

When reduced by sulphur dioxide, it yields *m*-benzenedisulphonimide, $NH(SO_2\cdot C_6H_4\cdot SO_2)_2NH$, crystallising in prisms with $2H_2O$; the *potassium* ($1\frac{1}{2}H_2O$), *sodium* ($6H_2O$), *silver*, and *potassium silver* salts are described.

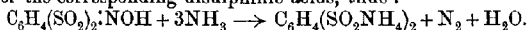
p-Benzenedisulphonehydroxamic acid (*p*-benzenedisulphonylhydroxylamine), $NOH(SO_2\cdot C_6H_4\cdot SO_2)_2NOH$, prepared by the action of nitrous acid on *p*-benzenedisulphonic acid, crystallises with $1\frac{1}{2}COMe_2$ in thin plates, and decomposes suddenly at 210° .

p-Benzenedisulphonimide, $NH(SO_2\cdot C_6H_4\cdot SO_2)_2NH$, crystallises with $2H_2O$ in colourless prisms; the *sodium*, *potassium*, *silver*, and *mercurous* salts are described.

p-Bromo-*o*-benzenedisulphonylhydroxamic acid, $C_6H_3Br(SO_2)_2\cdot NOH$, crystallises in prisms, and decomposes at 82° ; it is reduced by sulphur dioxide to the corresponding *sulphonimide*.

o-Benzenedisulphonylhydroxamic acid crystallises in colourless prisms, and decomposes at 45° ; when reduced, it yields a *sulphonimide*.

The benzenedisulphonehydroxamic acids described above are converted by cold fuming nitric acid into the corresponding sulphonimides, and are decomposed by aqueous ammonia, yielding ammonium salts of the corresponding disulphonic acids, thus:



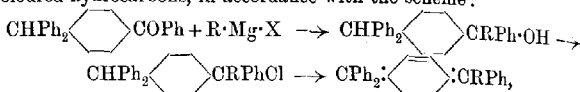
Ammonium m-benzenedisulphonate, $C_6H_4(SO_2NH_4)_2\cdot H_2O$, crystallises in star-like clusters of elongated tetrahedra, m. p. $113-115^\circ$; the *silver* salt, $C_6H_4(SO_2Ag)_2$, is crystalline. *Ammonium p-benzenedisulphonate*, $C_6H_4(SO_2NH_4)_2\cdot H_2O$, crystallises in slender needles, and decomposes at about 200° ; the *silver* salt is amorphous. W. H. G.

[Coloured and Colourless Triphenylmethyl.] BERNHARD FLURSCHHEIM (*Ber.*, 1908, **41**, 2746-2747).—Schmidlin's equilibrium

formula, $\text{CPh}_2\text{:CPh}_2 \rightleftharpoons \text{CPh}_3$, for triphenylmethyl (this vol, i, 623) has been already suggested by the author (Abstr., 1905, i, 614).

C. S.

Preparation of Hydrocarbons of the Quinodimethane Series and their Derivatives. ALEXEI E. TSCHITSCHIBABIN (*Ber.*, 1908, 41, 2770—2778. Compare Thiele and Balhorn, Abstr., 1904, i, 491; Staudinger, this vol., i, 410, 411).—The tendency of *p*-benzhydryltriphenylmethyl chloride or bromide to acquire an orange colour (this vol., i, 624) is due to the ready elimination of hydrogen halide with the formation of tetraphenylquinodimethane, $\text{CPh}_2\text{:} \langle \text{C}_6\text{H}_4 \rangle \text{:CPh}_2$. This reaction leads to a convenient method for the preparation of such coloured hydrocarbons, in accordance with the scheme:



where $\text{R} = p\text{-C}_6\text{H}_7$, $\alpha\text{-C}_{10}\text{H}_7$, or $p\text{-C}_6\text{H}_4\text{Br}$.

The elimination of the halogen acid occurs slightly at the ordinary temperature, but is best effected by quinoline in hot xylene in an atmosphere of carbon dioxide.

The converse operation, the addition of the halogen acid to the quinodimethane hydrocarbon, proceeds readily, resulting in the formation of the original halide, $\text{CHPh}_2 \langle \text{C}_6\text{H}_4 \rangle \text{CPh}_2\text{Cl}$, in the case of tetraphenylquinodimethane, whilst with unsymmetrical hydrocarbons the addition takes place in two directions, yielding the original halide, $\text{CHPh}_2 \langle \text{C}_6\text{H}_4 \rangle \text{CRPhCl}$, or the isomeride, $\text{CPh}_2\text{Cl} \langle \text{C}_6\text{H}_4 \rangle \text{CHPh}_2$; when $\text{R} = \alpha\text{-C}_{10}\text{H}_7$, both additive products are obtained.

Tetraphenylquinodimethane has the properties described by Thiele and Balhorn and by Staudinger (*loc. cit.*). The m. p. in a sealed capillary tube filled with carbon dioxide is 268° (partial decomp.). The addition of hydrogen chloride or bromide is effected by treating a solution of the hydrocarbon in hot benzene with the hydrogen halide, dissolved in glacial acetic acid, in an atmosphere of carbon dioxide.

p-Benzhydryltriphenylmethyl bromide does not melt at 140° as given (Tschitschibabin, *loc. cit.*), but decomposes gradually above 200° .

The reaction between magnesium α -naphthyl bromide and *p*-benzoyltriphenylmethane in ether leads, after the usual operations, to the formation of a viscous oil, which is converted by hydrogen chloride in glacial acetic acid into *p*-benzhydryldiphenyl- α -naphthylmethyl chloride, $\text{CHPh}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CPhCl} \cdot \text{C}_{10}\text{H}_7$, m. p. $129\text{--}130^\circ$, from which triphenyl- α -naphthylquinodimethane, $\text{CPh}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CPh} \cdot \text{C}_{10}\text{H}_7$, is obtained. The hydrocarbon has m. p. $240\text{--}241^\circ$ (in carbon dioxide), forms intensely orange-red crystals, and, like the chloride, dissolves in concentrated sulphuric acid with a green or violet-red colour, according to the concentration of the solution. The dry hydrocarbon is stable in air, but oxidises readily in solution. The addition of hydrogen chloride results mainly in the formation of *p*-phenyl- α -naphthylmethyltri-

phenylmethyl chloride, $\text{C}_6\text{H}_5\text{Cl} \cdot \text{C}_6\text{H}_4 \cdot \text{CHPh} \cdot \text{C}_{10}\text{H}_7$, m. p. 174—175° (decomp.).

p-Benzhydryldiphenyl-p-tolylmethyl bromide,
 $\text{CHPh}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CPhBr} \cdot \text{C}_7\text{H}_7$,
 m. p. 156—164° (decomp.), is a yellow powder. *Triphenyl-p-tolylquinodimethane*, $\text{CPh}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CPh} \cdot \text{C}_7\text{H}_7$, m. p. 197° (in carbon dioxide), forms orange-red crystals, develops an orange colour in sulphuric acid, and is very sensitive to oxygen or light when incompletely dried. *p-Bromotetraphenylquinodimethane*, $\text{CPh}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CPh} \cdot \text{C}_6\text{H}_4\text{Br}$, m. p. 251—259° (decomp.) in carbon dioxide, is a brownish-red, crystalline powder; it dissolves in sulphuric acid to an orange-red solution.
 C. S.

Sodium Derivative of Fluorene. RUDOLF WEISSGERBER (*Ber.*, 1908, 41, 2913—2916).—The reaction between fluorene and sodium or sodamide occurs at much lower temperatures than with potassium hydroxide (*Abstr.*, 1901, i, 521). When fluorene is heated with the calculated amount of sodamide at 120°, and finally at 150°, or with sodium at 180—200°, the sodium derivative, $\text{C}_6\text{H}_4 > \text{CHNa}$, is obtained as a hard, yellowish-brown, crystalline mass, which is decomposed by water, yielding fluorene and sodium hydroxide; bis-diphenylene-ethane is formed as a by-product. The same reaction occurs when dry ammonia is passed into fused sodium and fluorene at 150°, but in this case the by-products are very small.
 C. S.

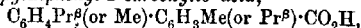
Structure of Retene. PAUL LUX (*Monatsh.*, 1908, 29, 763—777).—It has been stated by Fortner (*Abstr.*, 1904, i, 729) that retene is either 2-methyl-8-propyl- or 8-methyl-2-propyl-phenanthrene. This conclusion is confirmed by the present author, who finds that when retene ketone or retenequinone is fused with potassium hydroxide, a methylisopropyldiphenyl-2-carboxylic acid is formed; the position of the carboxyl group is decided by the fact that the acid cannot be esterified; hence one of the side-chains in retene is in the ortho-position to the $\text{CH}:\text{CH}$ group. At the same time a small amount of an isopropyldiphenyldicarboxylic acid is produced; as this readily yields a neutral ester, it is probably either 4-isopropyldiphenyl-2:3'- or 3-isopropyldiphenyl-2':4'-dicarboxylic acid.

Dimethyl fluorenonedicarboxylate, m. p. 188—189° (corr.) (Bamberger and Hooker, *Abstr.*, 1885, 906, give m. p. 184°), when partly hydrolysed, furnishes an acid ester, m. p. 260—261° (corr.), and by partly esterifying the dicarboxylic acid a second acid ester, m. p. 275—277° (corr.), is obtained. The silver salt of the first ester, on distillation in a vacuum, gives methyl fluorenone-2-carboxylate, m. p. 186—187° (corr.) (Fortner gives 181°), and the silver salt of the second ester yields the same compound, so that apparently an interchange of silver and methyl takes place; hence the constitution of the two acid esters cannot be decided from these data, and the intended preparation of fluorenone-1- and -2-carboxylic acids could not be effected.

Reteneglycollic acid is produced when retenequinone is boiled with

10% methyl-alcoholic potash; the *methyl* ester has m. p. 112–113° (corr.).

Methylisopropylidiphenyl-2-carboxylic acid,



prepared by fusing a mixture of retenequinone (or retene ketone) and lead peroxide with potassium hydroxide, crystallises in octahedra, m. p. 132–134° (corr.); the *silver* salt was analysed. By treating the acid with thionyl chloride, or by distilling it with lime or its barium salt with sodium methoxide, retene ketone is produced. On distilling the silver salt in a current of hydrogen, the acid passes over.

4-isopropylidiphenyl-2:3'-dicarboxylic acid (or *3-isopropylidiphenyl-2':4'-dicarboxylic acid*) has m. p. 247–249° (corr.); the *methyl* ester is a white, microcrystalline powder, m. p. 93–95° (corr.); the *silver* salt was analysed.

J. C. C.

Nitration of Aniline and its Derivatives. OTTO N. WITT and ERHARD WITTE (*Ber.*, 1908, 41, 3090–3097).—In continuation of the investigation of Witt and Utermann (compare Abstr., 1907, i, 27), the effect produced by using a very large excess of strong mineral acids during the nitration of aniline has been studied. Both *o*- and *p*-nitroaniline, dissolved in concentrated sulphuric acid (monohydrate), are converted by a mixture of concentrated sulphuric and nitric acids (prepared by adding dry potassium nitrate to the sulphuric acid), at temperatures below 5°, into picramide, the yield being between 58% and 67% of the theoretical, and trinitrophenylnitramic acid. Picramide may also be prepared in a similar manner from acetanilide, the yield being 67% of the theoretical.

m-Nitroaniline is converted by the nitric-sulphuric acid mixture into tarry products, but *m*-nitroacetanilide under the same treatment yields 3:4-dinitroacetanilide; if the action is allowed to proceed for several days, this substance undergoes further nitration, forming 3:4:6-trinitroaniline.

The conclusion drawn by Martinsen (Abstr., 1905, ii, 149), that only 67% of the *p*-nitroaniline is converted into picramide because of a simultaneous sulphonation of the *p*-nitroaniline, is thus shown to be incorrect, since the remainder is converted into trinitrophenylnitramic acid; further, picramide is not best prepared by the nitration of aniline in the presence of sulphuric acid, as stated by this author, since the *m* nitroaniline formed together with the ortho- and para-isomerides would not be converted into picramide.

Picramide acetate, $\text{C}_8\text{H}_4\text{O}_7\text{N}_4$, prepared by adding one drop of concentrated sulphuric acid to a solution of picramide in acetic anhydride, forms colourless needles, which commence to decompose at 207°, m. p. about 230°.

The following additive compounds of picramide (compare Mertens, Abstr., 1878, 725) are prepared by boiling equivalent quantities of the constituents dissolved in acetic acid. *Picramide α -naphthylamine*, $\text{C}_6\text{H}_4\text{O}_6\text{N}_4\cdot\text{C}_{10}\text{H}_9\text{N}$, crystallises in dark red needles, having a metallic lustre; the *β -naphthylamine additive* compound forms red crystals.

Picramide α -naphthol, $\text{C}_6\text{H}_4\text{O}_6\text{N}_4\cdot\text{C}_{10}\text{H}_8\text{O}$, crystallises in scarlet needles; the *β -naphthol isomeride* crystallises in red needles.

Sodium trinitrophenylnitramate, $\text{Na} \begin{array}{c} \text{NO}_2 \\ | \\ \text{N} \cdot \text{C} \end{array} \begin{array}{c} \text{C}(\text{NO}_2) \cdot \text{CH} \\ \text{C}(\text{NO}_2) \cdot \text{CH} \end{array} \text{C} \cdot \text{NO}_2$,
 crystallises in yellow needles, and decomposes violently at 203° .
 3:4:6-Trinitroaniline, $\text{C}_6\text{H}_4\text{O}_6\text{N}_4$, crystallises in pale yellow needles. W. H. G.

Reduction of Aromatic Nitro-compounds by Sodium Sulphide. JAN J. BLANKSMA (*Proc. K. Akad. Wetensch. Amsterdam*, 1905, 11, 42—43. Compare Abstr., 1900, i, 226, 482; 1901, i, 246, 460, 461).—The author has shown previously that when sodium disulphide acts on aromatic nitro-compounds containing halogen atoms or nitro-groups, the latter, when in the ortho- or para-position to a nitro-group, are replaced by the group S_2 , forming disulphides. When no such atoms or groups are present, amino-compounds are formed together with small amounts of azoxy-compounds. It is now found that, when reduced with sodium disulphide in alcoholic solution, nitrobenzene, *o*- and *m*-nitrotoluenes, *o*-, *m*-, and *p*-nitroanisoles, *m*-chloro- and -bromo-nitrobenzenes, and 3:5-dichloro- and -dibromo-nitrobenzenes give a 70% yield of the corresponding amines. In the case of *o*- and *p*-chloronitrobenzenes, where the halogen atom is replaced by the group S_2 , small amounts of *o*- and *p*-chloroanilines are formed. *p*-Nitrotoluene gives *p*-aminobenzaldehyde in addition to *p*-toluidine. By partial reduction, 3:5-dinitrotoluene furnishes 5-nitro-*m*-toluidine, 3:5-dinitroanisole gives 5-nitro-*m*-anisidine, 2:4-dinitroanisole (or phenetole) yields 4-nitro-*o*-anisidine (or phenetidine), and 1:3:5-trinitrobenzene gives 3:5-dinitroaniline. A small quantity of the azoxy-compounds is also usually produced. J. C. C.

Preparation and Constitution of Sulphoxides and Disulphoxides. OSCAR HINSBERG (*Ber.*, 1908, 41, 2836—2839).—Organic sulphides are converted by hydrogen peroxide into sulphoxides; disulphides under similar conditions take up two oxygen atoms, forming disulphoxides. This reaction has been proved to occur in the case of benzyl, phenyl, and β -naphthyl disulphides. Reasons are stated for adopting the formula $\text{R} \cdot \text{SO} \cdot \text{SO} \cdot \text{R}$ for the disulphoxides.

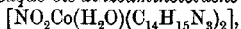
pp-Diacetyldiaminodiphenyl sulphoxide, $\text{SO}(\text{C}_6\text{H}_4 \cdot \text{NHAc})_2$, crystallises in small plates, m. p. 278° . The conversion of disulphide into disulphoxide is best carried out in cold acetic acid solution, using an excess of 3% hydrogen peroxide. E. F. A.

Compounds of Cobaltinitrites with *p*-Toluidine, Diazoaminotoluene, Hydrazine, and Nitrosohydrazine. KARL A. HOFMANN and KARL BUCHNER (*Ber.*, 1908, 41, 3084—3090).—It has been shown by Hofmann and Burger (Abstr., 1907, i, 751) that ammonium cobaltinitrite and ethylammonium cobaltinitrite decompose when heated, the component parts interacting in a normal manner. Such reactions between the nitrite group of the complex and that part of the salt outside the complex are of great interest, in that they throw light on the mode of union of the latter with the complex; further, since the nitrite group attached to the cobalt atom is not very

reactive, the formation of substances was to be expected, which would ordinarily be produced so rapidly as intermediate products when free nitrous acid is employed as to render their isolation impossible.

p-Toluidine reacts with sodium cobaltinitrite in aqueous-alcoholic solution containing acetic acid, yielding diazoaminotoluene and *p*-toluidine tetranitrito-bis-*p*-toluidinocobaltiate, $[\text{N}_4\text{O}_8\text{Co}(\text{C}_6\text{H}_4\text{N})_2]\text{H}_2\text{C}_6\text{H}_4\text{N}$, crystallising in brownish-red, pointed, compact prisms. It is converted by alcoholic ammonia into ammonium cobaltinitrite, with elimination of *p*-toluidine. It is stable at the ordinary temperature, but decomposes when its aqueous solution is boiled, yielding nitroresol with evolution of nitrogen.

In the absence of acetic acid, *p*-toluidine reacts with sodium cobaltinitrite, forming *nitritouquo-bis-diazoaminotoluene cobalt*,



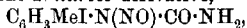
which crystallises in glistening, black, thick, rhombic plates or pyramids. It is very stable towards acids and alkalis, being attacked by these only at high temperatures.

Hydrazine unites with sodium cobaltinitrite in solutions containing small quantities of acetic acid, yielding *sodium diammonium cobaltinitrite*, $[\text{Co}(\text{NO}_2)_6]\text{NaN}_2\text{H}_4$, which forms yellow, rectangular crystals. It decomposes with evolution of two atoms of nitrogen when its aqueous solution is heated, and does not explode when heated alone. It loses $2\text{H}_2\text{O}$ when kept for some time over concentrated sulphuric acid, yielding *sodium nitrosohydrazine cobaltinitrite (sodium nitrosohydrazinetetranitritocobaltiate)*, $[\text{N}_4\text{O}_8\text{Co}(\text{NO}\cdot\text{NH})_2]\text{Na}$, obtained as glistening, dark purplish-violet crystals.

Hydroxodinitritohydrazine cobalt, $[\text{HO}(\text{N}_2\text{O}_4)\text{Co}(\text{N}_2\text{H}_4)\text{NH}_2]_2$, is formed by the action of an alkaline solution of hydrazine on sodium cobaltinitrite. It forms aggregates of dark purplish-red needles, decomposes violently when heated, and liberates nitrogen when boiled with water.

W. H. G.

Derivatives of 2-Iodo-*p*-toluidine and 2-Iodo-4-nitrobenzoic Acid containing Uni- and Ter-valent Iodine. CONRAD WILGERODT and RUDOLF GARTNER (*Ber.*, 1908, 41, 2813—2826).—I. *2-Iodo-*p*-toluidine and its Derivatives.*—*2-Iodo-*p*-toluidine*, $\text{C}_6\text{H}_4\text{MeI}\cdot\text{NH}_2$, prepared by reducing 2-iodo-*p*-nitrotoluene with ferrous sulphate in ammoniacal solution, crystallises in large, white needles, m. p. 37° ; the *hydrochloride*, long, rather dark needles, the *sulphate*, shining leaflets, the *nitrate*, well-formed rhombs, and the *oxalate*, small rhombs, are described. *2-Iodoaceto-*p*-toluidide*, $\text{C}_6\text{H}_4\text{MeI}\cdot\text{NHAc}$, prepared from the base and acetic anhydride, forms white needles, m. p. 130° . *2-Iodo-*p*-tolylcarbamide*, $\text{C}_6\text{H}_4\text{MeI}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$, forms well-defined rhombs, m. p. 194° ; the *N-nitroso-derivative*,

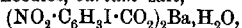


crystallises in yellow, shining needles, which soon turn brown and decompose in the air, m. p. 99° (decomp). *Aceto-*p*-toluidide o-iodo-chloride*, $\text{C}_6\text{H}_3\text{Me}(\text{ICl}_2)\cdot\text{NHAc}$, prepared by passing chlorine into a glacial acetic acid solution of 2-iodoaceto-*p*-toluidide to which chloroform is added, forms yellow needles, decomposing at 100° . It was not found possible to prepare 2-iodoso- or 2-iodoxy-aceto-*p*-toluidide. An aqueous

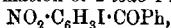
solution of *phenyl-p-acetylaminotolyliodonium hydroxide*,
 $\text{C}_6\text{H}_5 \cdot \text{I}(\text{OH}) \cdot \text{C}_6\text{H}_4\text{Me} \cdot \text{NHAc}$,

is obtained by the interaction of the iodochloride and mercury diphenyl; the *iodide*, pale yellow needles, m. p. 145°, the *bromide*, white prisms, m. p. 159.5–160°, the *dichromate*, yellow needles, becoming reddish-brown when dry and decomposing at 80°, and the *platinichloride*, small, yellow crystals, m. p. 125° (decomp.), are described.

II. *2-Iodo-4-nitrobenzoic acid and its Derivatives*.—*2-Iodo-4-nitrobenzoic acid*, prepared by oxidising 2-iodo-*p*-nitrotoluene with nitric acid under pressure, forms long, pale yellow needles, m. p. 142°; the *silver salt*, white needles, *barium salt*,

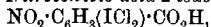


methyl ester, long needles, m. p. 89°, and *ethyl ester*, large, shining prisms, m. p. 44°, are described. *2-Iodo-4-nitrobenzoyl chloride* forms yellow needles, b. p. 196°/18 mm.; the *amide* forms yellow, rhombic crystals, m. p. 205°. The interaction of 2-iodo-4-nitrobenzoyl chloride, benzene, and aluminium chloride in presence of a small quantity of light petroleum leads to the formation of *2-iodo-4-nitrobenzophenone*,



which forms aggregates of small needles, m. p. 90–91°; on heating with hydroxylamine hydrochloride and a little alcohol under pressure, there is formed the *oxime* in small prisms, m. p. 161–161.5°. This, when boiled with 50% aqueous sodium hydroxide, is condensed to

5-nitrophenylindoxazen, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \begin{smallmatrix} \text{CPh} \\ \diagup \quad \diagdown \\ \text{O} \end{smallmatrix} \text{N}$, which forms small rhombs, m. p. 135°. *4-Nitrobenzoic acid 2-iodochloride*,



prepared by the action of chlorine on a chloroform solution of 2-iodo-4-nitrobenzoic acid, forms yellow needles; on warming with 10% aqueous sodium hydroxide, it is converted into *2-iodoso-4-nitrobenzoic acid*, $\text{NO}_2 \cdot \text{C}_6\text{H}_3(\text{IO}) \cdot \text{CO}_2\text{H}$ or $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \begin{smallmatrix} \text{CO} \\ \diagup \quad \diagdown \\ \text{I}(\text{OH}) \end{smallmatrix} \text{O}$, which is also

formed in small amount by treating 2-iodo-4-nitrobenzoic acid with fuming nitric acid. This acid crystallises in white needles, the m. p. of which varies with different preparations; the m. p.'s 190°, 196°, and 201° were observed. It can be crystallised unchanged from formic acid, and, when boiled with aqueous sodium hydroxide and alcohol, is decomposed with formation of iodic, 2-iodo-4-nitrobenzoic, and 4-nitrobenzoic acids. The *sodium salt*, brown leaflets, *silver salt*, small needles, *barium salt*, yellow needles, *copper salt*, bright green, amorphous precipitate, and *lead salt*, yellow powder, are described. *Methyl 2-iodo-4-nitrobenzoate*, when treated with chlorine in chloroform solution, yields *methyl-4-nitrobenzoate-2-iodochloride* as a pale yellow, crystalline precipitate, which, under the action of sodium hydroxide or carbonate, gives 2-iodoso-4-nitrobenzoic acid. The *methyl ester* of 2-iodoso-4-nitrobenzoic acid was finally prepared by the action of hydrogen chloride on a methyl-alcoholic solution of 2-iodoxy-4-nitrobenzoic acid (see below); it crystallises in small needles, m. p. 180–181°.

2-Iodoxy-4-nitrobenzoic acid, $\text{NO}_2 \cdot \text{C}_6\text{H}_3(\text{IO}_2) \cdot \text{CO}_2\text{H}$, prepared by

oxidising the iodoso-acid with potassium permanganate in sulphuric acid solution, or, better, with sodium hypochlorite, forms white needles, m. p. 206° (mild explosion); the *silver* salt, small needles, which explode on heating, and the *lead* salt, bright yellow powder, are described.

III. *Preparation of 2-Iodo-4-aminobenzoic Acid.*—2-Iodo-4-acetylaminobenzoic acid, prepared by oxidising 2-iodoaceto-*p*-toluidide with potassium permanganate with the addition of magnesium sulphate, forms needles, m. p. 213—214°. On hydrolysis with concentrated hydrochloric acid, it yields 2-iodo-4-aminobenzoic acid, crystallising in needles, m. p. 180° (decomp.), which can also be obtained by reducing 2-iodo-4-nitrobenzoic acid with stannous chloride and hydrochloric acid; the *hydrochloride*, well-formed prisms, the *silver* salt, small, white needles, and the *methyl* ester, needles, m. p. 112°, are described.

J. C. C.

Quaternary Phenacylammonium Salts. EDGAR WEDEKIND (*Ber.*, 1908, 41, 2802—2805).—Quaternary phenacylammonium salts are obtained when one molecule of a tertiary base is allowed to act on one molecule of α -bromoacetophenone at the ordinary temperature, care being taken that when the bromide is dissolved in the tertiary base the temperature does not exceed 30—40°.

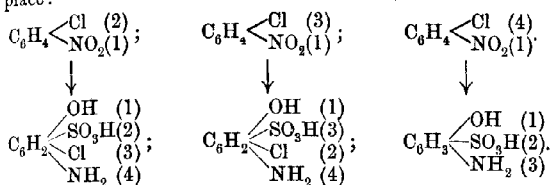
Phenacylphenyldimethylammonium bromide (Abstr., 1902, i, 278) sinters between 100° and 110°, and decomposes between 115° and 125°. By shaking with moist silver oxide, it yields the corresponding *base*, from which the *platinichloride*, small, yellow prisms, and the *aurichloride* were prepared. Phenacylphenylbenzylmethylammonium bromide crystallises in fine, colourless needles, which decompose at 149—150°. Phenacylphenylmethylethylammonium bromide forms crystals, decomposing at 145°; the *d*-bromocamphorsulphonate is not suitable for experiments on resolving the substance, and no resolution could be effected by means of the *d*-camphorsulphonate. The latter forms small, colourless crystals, decomposing at 176°.

J. C. C.

Phenylcarbylamine from Pyrogallol and Nitrobenzene. HEINRICH BRUNNER and V. VUILLEUMIER (*Chem. Zentr.*, 1908, ii, 588; from *Schweiz. Woch. Chem. Pharm.*, 1908, 46, 436).—When a few drops of nitrobenzene are heated with a small quantity of pyrogallol in an aqueous alkaline solution, the characteristic odour of phenylcarbylamine is strongly developed. In alcoholic solution, the reaction proceeds differently, ammonia being evolved and no formation of carbylamine taking place. If, however, the product is treated with ether and fractionated in a vacuum, carbylamine may be isolated. The use of catechol instead of pyrogallol, leads to the formation of only a trace, whilst with resorcinol, quinol, phloroglucinol, and hydroxyquinol, carbylamine is not produced. *o*-, *m*-, and *p*-Chloro- and bromo-nitrobenzenes and *o*- and *m*-nitrotoluenes give carbylamine, but nitromethane, nitroethane, nitropropane, dinitrobenzene, *o*- and *p*-nitrophenols, β -naphthol, and *p*-nitrotoluene liberate ammonia, and do not give carbylamine.

J. V. E.

***p*-Aminophenolsulphonic Acid.** HEINRICH BRUNNER and V. VUILLEUMIER (*Chem. Zentr.*, 1908, ii, 587—588; from *Schweiz. Woch. Chem. Pharm.*, 1908, 46, 434—436).—When nitrobenzene is heated alone with sulphuric acid at 225°, there is obtained a small quantity of *p*-aminophenolsulphonic acid. In a similar manner, when resorcinol, quinol, catechol, pyrogallol, glycerol, mannitol, or dextrose is heated with nitrobenzene and sulphuric acid, 4-aminophenol-2-sulphonic acid is produced. This may be explained by an oxygen atom of the nitrobenzene wandering to the para-position, and then reduction occurring by the sulphur dioxide produced from the sulphuric acid and phenol. This view is supported by the fact that nitrobenzene, when heated with carbon and sulphuric acid, yields *p*-aminophenolsulphonic acid. Wandering of oxygen appears to be a common property of the nitro-group, as is indicated by the following observed changes which take place:



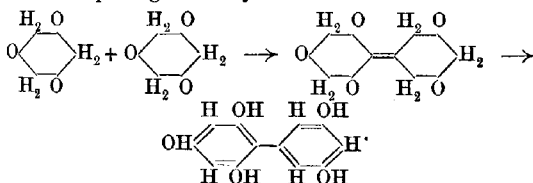
The corresponding bromo-derivatives behave in a similar manner.

J. V. E.

Picrates. OTTO ANSELMINO (*Ber.*, 1908, 41, 2996).—When picric acid or yellow ammonium picrate is dissolved in 10% ammonium hydroxide at 80°, and crystallisation is induced at a higher temperature, a red modification of ammonium picrate, $\text{C}_6\text{H}_5\text{O}_7\text{N}_4$, is obtained, which appears to be stable when kept, but reverts to the yellow form on recrystallisation (compare Suida, this vol., i, 523).

C. S.

Phloroglucide. JOSEPH HERZIG and RICHARD KOHN (*Monatsh.*, 1908, 29, 677—688).—The ease with which phloroglucide is formed from 2 mols. of phloroglucinol by loss of 1 mol. of water under the



influence of concentrated sulphuric acid suggests that the glucide is an anhydride of the glucinol. Herzig, however, found (*Abstr.*, 1899, i, 31) that phloroglucide forms a penta-acetyl derivative, and considered that the two phloroglucinol residues must be united by a carbon linking. A further study of phloroglucide has now confirmed the presence of the five hydroxyls, and it is suggested that the

formation of the glucide may take place according to the preceding scheme.

When treated with diazomethane in ethereal solution, or with methyl sulphate and potassium hydroxide, phloroglucide forms a *pentamethyl ether*, $C_{12}H_5(OMe)_5$, which separates from alcohol in white crystals, m. p. 117–120°. The *monomethyl ether*, $C_{12}H_5O_4OMe$, prepared by the action of hydrogen chloride on the glucide in methyl-alcoholic solution, has m. p. 222–225°, yields the preceding pentamethyl ether on treatment with diazomethane, and, when boiled with acetic anhydride and sodium acetate, forms a *tetra-acetyl derivative*, $OMe \cdot C_{12}H_5(OAc)_4$, which is obtained in colourless crystals, m. p. 102–105°. The *monosthyl ether*, $C_{12}H_5O_4 \cdot OEt$, has m. p. 165–168°.

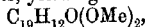
Pentabromophloroglucide, $C_{12}Br_5(OH)_5$, formed by treating phloroglucide with bromine in glacial acetic acid or dichloroethylene solution, separates from ethyl acetate in colourless crystals, m. p. 242–245°, yields with diazomethane a *pentamethyl ether*, $C_{12}Br_5(OMe)_5$, m. p. 200–202°, and, when boiled with acetic anhydride and sodium acetate, forms a *penta-acetyl derivative*, $C_{12}Br_5(OAc)_5$, m. p. 198–202°.

Bromination of pentamethylphloroglucide in dichloroethylene solution leads to the formation of a *tetrabromo-derivative*, $C_{12}HBr_4(OMe)_5$, which separates from ethyl acetate in colourless crystals, m. p. 228–229°.

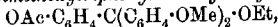
The tautomeric nature of phloroglucide appears in that, whilst the bromine of the pentabromophloroglucide is eliminated quantitatively by means of potassium hydroxide, the brominated ethers are stable under the same conditions. The results of a study of the hydrolysis of the tetra-acetyl derivative of monomethylphloroglucide by means of sulphuric acid show that with very dilute acid the four acetoxy groups are hydrolysed completely, but that with increasing concentration of the acid there appears a condensation in which one acetyl group takes part; this reaction is smoothest when the proportion of acid to water present is 3 : 1.

G. Y.

Aurin Dimethyl Ether. JOSEF HERZIG (*Monatsh.*, 1908, 29, 653–659. Compare Baeyer and Villiger, *Abstr.*, 1902, i, 769).—Aurin is readily methylated by diazomethane, although not by other common methylating agents, yielding *aurin dimethyl ether*,



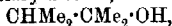
which crystallises in orange needles, m. p. 183–186°, is soluble in aqueous alkalis, and, when recrystallised from dilute hydrochloric acid, is converted into a *hydrate*, $C_{21}H_{18}O_8 \cdot H_2O$; this forms red crystals, m. p. 57–65°, and loses H_2O at 110°, forming again the dimethyl ether, m. p. 183–186°. Whilst aurin is readily acetylated, forming tri-acetylaurincarbinol, aurin dimethyl ether reacts only with difficulty with acetic anhydride alone or in presence of sodium acetate, but, on treatment with the anhydride and sulphuric acid, it yields a white *product*, which, on recrystallisation from alcohol, forms the *acetyl derivative*, *acetoxydimethoxytriphenylcarbinyl ethyl ether*,



crystallising in white needles, m. p. 136–138°. The *acetyl derivative* of trimethoxytriphenylcarbinol, $C_{19}H_{15}(OMe)_3 \cdot OAc$, prepared by recrystallisation of the white product from methyl alcohol, forms

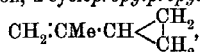
white crystals, m. p. 105–108°. Both acetyl derivatives readily undergo partial hydrolysis, becoming slightly yellow. It is considered that the immediate product of the acetylation is probably the diacetyl derivative, $C_{13}H_{12}(OMe)_2(OAc)_2$. G. Y.

Relative Stability of Cyclic Polycarbon Groups. LOUIS HENRY (*Compt. rend.*, 1908, 147, 557–561).—In an earlier paper (*Abstr.*, 1907, i, 374), the author has shown that, under the action of suitable reagents, the tertiary alcohol, dimethylisopropylcarbinol,



loses H_2O , and the corresponding bromide loses HBr , to form the isomeric ethylenic hydrocarbons, $CMe_2 : CMe_2$ and $CHMe_2 : CMe : CH_2$, in the proportions of 3 : 1. The investigation has been extended to cyclopropyldimethylcarbinol, $OH \cdot CMe_2 \cdot CH \begin{smallmatrix} \diagup CH_2 \\ \diagdown CH_2 \end{smallmatrix}$, and the results

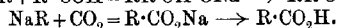
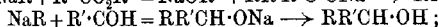
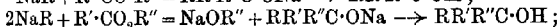
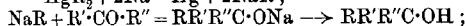
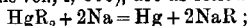
show that the cyclic group, $-CH \begin{smallmatrix} \diagup CH_2 \\ \diagdown CH_2 \end{smallmatrix}$, is much more stable than the isopropyl group, $-CHMe_2$; thus, when cyclopropyldimethylcarbinol is treated with acetic anhydride, or its bromide is heated with potassium acetate, the acetate, $OAc \cdot CMe_2 \cdot CH \begin{smallmatrix} \diagup CH_2 \\ \diagdown CH_2 \end{smallmatrix}$, b. p. 159–160°, is formed; the ethylenic hydrocarbon, α -cyclopropylpropylene, b. p. 77°,



is only formed when the alcohol is heated with phosphoric oxide, or the bromide heated at 170° with powdered potassium hydroxide.

M. A. W.

Sodium-alkyl Compounds and Syntheses made Therewith. PAUL SCHORIGIN (*Ber.*, 1908, 41, 2717–2723).—The author has investigated the action of a mixture of sodium and mercury dialkyls (which probably contains the corresponding sodium dialkyl; compare Buckton, *Annalen*, 1859, 112, 320) on carbon dioxide, ketones, aldehydes, and esters. The reactions, which confirm the author's assumption that sodium alkyls are formed in syntheses by means of sodium and alkyl halides (this vol., i, 866), are as follows:



When an ethereal solution of mercury diethyl and benzophenone is added to ether and sodium, a stream of nitrogen or hydrogen being led through the mixture, a vigorous reaction occurs, with the production of diphenylethylcarbinol in a 40% yield. The α -diphenylpropylene obtained from this has m. p. 48.5° (Masson, *Abstr.*, 1903, i, 28, gives 51°; the author states that Masson gives 6°, but this is the m. p. ascribed by the latter to diphenylethylene. Klages, *Abstr.*, 1902, i, 668, gives 52°, as do also Hell and Bauer, *Abstr.*, 1904, i, 241). Sodium, mercury diethyl, and methyl benzoate give a 24% yield of

phenyldiethylcarbinol, D_4^0 0.9986, D_4^{10} 0.9836, b. p. $109^\circ/15$ mm. (Kling, Abstr., 1904, i, 2, gives $125-127^\circ/10$ mm.; Grignard, Abstr., 1904, i, 214, gives $101-102^\circ/11$ mm.; Klages, Abstr., 1904, i, 28, gives $107-109^\circ/14$ mm., and $223-224^\circ$ under atmospheric pressure).

By the interaction of sodium, mercury diethyl and benzaldehyde, phenylethylcarbinol is produced in a 15% yield, b. p. $108-109^\circ/17$ mm.; the density varies from D_4^1 1.017 to 1.020, probably owing to the presence of benzyl alcohol (Wagner, *J. Russ. Phys. Chem. Soc.*, 1884, 16, 322, gives b. p. 212° , D_4^0 1.016; Errara, Abstr., 1887, 35, gives b. p. $215-217^\circ$). A specimen prepared from magnesium ethyl iodide and benzaldehyde had b. p. $108.5^\circ/17$ mm., and D_4^1 1.012-1.015. The identity of both preparations was proved by oxidising them to propiophenone, which yielded an identical semicarbazone, m. p. 175° (corr.) (Blaise, Abstr., 1902, i, 164, gives 182°).

By passing a current of carbon dioxide into a mixture of sodium and mercury diethyl in ethereal solution, propionic acid is formed; when mercury dimethyl is used, acetic acid is produced, and by employing mercury diisocamyl, isohexioic acid results. J. C. C.

Lupeol. N. H. COHEN (*Arch. Pharm.*, 1908, 246, 520-522).—It is pointed out that the data recorded by Klobb (Abstr., 1903, i, 165) for antheasterol and its derivatives are in close agreement with those observed for lupeol and the corresponding derivatives of this (Likiernik, Abstr., 1891, i, 551, 1446; Romburgh, 1904, i, 905, and Cohen, 1907, i, 211), and that, consequently, antheasterol may be lupeol.

The same considerations applied to the phytosterol acetate obtained by Marino Zuco (Abstr., 1890, i, 757) from Dalmatian insect flowers, led to the supposition that this might be a mixture of acetates of amyirin and lupeol, but experiments made by the author, whilst giving slightly higher melting points for the various derivatives than those recorded by Marino Zuco, show that the substance contains neither lupeol nor amyirin. T. A. H.

Liquid Crystals of Ergosteryl Esters. PAUL GAUBERT (*Compt. Rend.*, 1908, 147, 498-500).—A series of esters prepared from Tanret's ergosterol and fongisterol (this vol., i, 637) has been examined with a view to ascertaining whether, like the esters of the closely-allied cholesterol, they yield liquid crystals (Lehmann, Abstr., 1906, i, 952, and Gaubert, 1907, ii, 939).

Ergosteryl esters present a liquid, anisotropic phase, but this is very difficult to observe in the cases of the acetate and propionate. The crystalline liquids are more viscous than those of the corresponding cholesteryl esters. Ergosterol, cholesterol, and fongisterol, when fused with orcinol, each yield a new compound presenting a liquid, crystal phase. Both ergosterol and fongisterol, when allowed to solidify on a glass slip under a cover glass, do not form spherulites with helical scrolls; this serves to distinguish them from cholesterol and phytosterol. T. A. H.

Double Linkings in Phytosterol and Cholesterol. ETTORRE MOLINARI and P. FENAROLI (*Ber.*, 1908, 41, 2785-2788).—Windaus'

supposition, that phytosterol contains two double linkings and cholesterol one (Abstr., 1907, i, 921), is not supported by Willstätter and Mayer (this vol., i, 636) or Langheld (*ibid.*, i, 316), whose results indicate the presence of two double linkings in cholesterol. The authors have therefore examined the behaviour of phytosterol from Calabar beans and nut oil, of cholesterol, and of bombycesterol in chloroform towards ozonised air; in each case an ozonide, $C_{27}H_{48}O_7$, is obtained, the ozone number proving the presence of two double linkings. *Phytosterol ozonide* is a white, crystalline substance, which decomposes at about 100° . The ozone number of ozonides which do not decompose at 50 – 60° can be determined in two to three hours by saturating the unsaturated compound in a volatile solvent with ozonised air and evaporating the solution to constant weight in a vacuum at 60° on the water-bath.

The authors regard the ozone number as the best indication of the number of true double linkings in an organic compound.

C. S.

Phytosterol from Cotton-seed Oil. A. HEIDUSCHKA and H. W. GLOTH (*Pharm. Zentr.-h.*, 1908, 49, 836–837).—The phytosterol of cotton-seed oil, prepared by saponifying the latter and extracting a solution of the soap in water with ether, when acetylated and then brominated as described by Windaus and Hauth (Abstr., 1907, i, 129, 921) does not, like stigmasterol (*loc. cit.*), deposit a crystalline acetate tetrabromide, but only a small quantity of an amorphous *dibromo-acetate* (m. p. 127° , approx.). The substance remaining in solution in the mother liquor is also a *dibromo-derivative*, $C_{29}H_{48}O_2Br_2$, and can be crystallised from alcohol. On treatment with zinc dust and sodium amalgam, it yields the corresponding acetate, and this, on hydrolysis, gives an alcohol, having m. p. 136° , identical with that of the phytosterol started with, so that the latter may be regarded as a single substance.

T. A. H.

Phytosterols from Balata. N. H. COHEN (*Arch. Pharm.*, 1908, 246, 510–514).—A continuation of work on the crystalline constituents of the portion of balata soluble in alcohol (Abstr., 1907, i, 715; compare Tschirch and Schereschewski, Abstr., 1905, i, 713). After the removal of β -amyrin acetate, the residue by crystallisation from alcohol yields two impure products, which can be separated into the same two constituents by the use of (1) mechanical means, followed by (2) fractional crystallisation. The first substance, identical with the β -balalban of Tschirch and Schereschewski (*loc. cit.*), $C_{31}H_{50}O_2$ or $C_{32}H_{52}O_2$, m. p. 111 – 112° , $[\alpha]_D + 57^\circ$ in chloroform, crystallises in colourless leaflets, and, on hydrolysis, furnishes an acid, having the reactions of formic acid, and a crystalline alcohol, m. p. 115 – 116° . The latter yields an *acetate*, m. p. 107 – 107.5° (which does not depress the m. p. of the original ester, so that the latter may be the acetate, not the formate), and a *benzoate*, m. p. 120 – 122.5° , crystallising from alcohol in lustrous leaflets.

The third *constituent*, m. p. 208 – 210° , crystallises in lustrous leaflets, and, on hydrolysis, yields lupeol (compare Abstr., 1907, i,

211) and an acid, having the odour of butyric acid, but the silver salt of which contains silver equivalent to that of silver acetate. *Lupeol butyrate*, m. p. 194—195°, crystallises in small, glancing leaflets, and depresses the melting point of the original ester when mixed with it. *Lupeol acetate*, m. p. 215°, does not depress the melting point of the original ester when mixed with it, but as the synthetic ester crystallises in needles and the natural one in leaflets, it is suggested that the latter may be the acetate of an isomeride of lupeol, which is converted into lupeol in the process of hydrolysis, or again, the difference in crystalline form may be due to the presence of an impurity in the natural ester, as prepared from balata.

T. A. H.

Phytosterols from South African "Rubber." N. H. COHEN (*Arch. Pharm.*, 1908, 246, 515—520).—The coagulated latex from a South African *Euphorbia*, which contained 70.0% of resin and only 5.5% of a caoutchouc-like substance, has been systematically examined with a view to the identification of the resin-alcohols it contains.

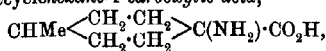
An extract of the product in boiling alcohol deposits, first, β -amyirin acetate, and eventually a second substance, $C_{30}H_{50}O$, m. p. 112—116°, $[\alpha]_D + 8.75^\circ$ in chloroform, which crystallises from cold alcohol in thin, lustrous, colourless leaflets, and is not affected by boiling with potassium hydroxide in alcohol. With acetic anhydride, it furnishes an *acetate*, m. p. 117—122°, $[\alpha]_D + 25^\circ$ in chloroform, but when heated with benzoyl chloride it yields two crystalline products, m. p. 146—152° and m. p. 159—164°, and these, on hydrolysis, furnish substances of m. p. 110.5—111° and 115—117.5° respectively, indicating possibly that the parent alcohol, m. p. 110°, may have been a mixture of two substances.

For the detection of lupeol (compare Abstr., 1907, i, 211), the portion of the coagulated latex soluble in alcohol was treated with potassium hydroxide in alcohol, and the neutral hydrolytic product benzoylated. No lupeol benzoate was found, but, in addition to amyirin benzoate, a second substance, $C_{31}H_{54}O$ or $C_{33}H_{56}O$, m. p. 193—195°, $[\alpha]_D + 75^\circ$ in chloroform, crystallising from acetone in glancing needles, was obtained. This, on hydrolysis by alkalis in alcohol, gave a *product*, $C_{24}H_{40}O.H_2O$ or $C_{26}H_{44}O.H_2O$, $[\alpha]_D + 58.6^\circ$, which crystallises in needles from acetone diluted with water, and in some respects resembles Schulze's *ischolesterol* (Abstr., 1898, i, 463), but, whilst the latter, according to Schulze, gives an amorphous acetate, the phytosterol from this *Euphorbia* latex yields a crystalline acetate, $C_{26}H_{42}O_2$ or $C_{28}H_{46}O_2$, m. p. 134—135°.

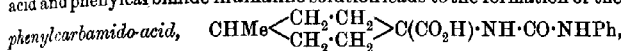
T. A. H.

Hydrocyclic α -Amino-acids. ALADAR SKITA and R. LEVI (*Ber.*, 1908, 41, 2925—2937).—The comparison of aliphatic and alicyclic α -amino-acids is not without interest, since recent researches (Zelinsky and Gutt, this vol., i, 638; Merling, *ibid.*, i, 653) have shown not unimportant differences in the behaviour of aliphatic and alicyclic carboxylic acids. Whilst the esters of *cis*-hydrocyclic δ -amino-acids yield unimolecular anhydrides by treatment with sodium ethoxide (Skita, Abstr., 1907, i, 1040), a similar behaviour of hydrocyclic α -amino-acids is impossible, since the amino- and the carboxyl groups,

being attached to the same carbon atom, must always have the *trans*-configuration. The esters of such amino-acids ought to yield bimolecular anhydrides, cyclic 2:5-diketopiperazines, which should hydrolyse to cyclic dipeptides. Such behaviour has been observed in the cases of 4-amino-1-methylcyclohexane-4-carboxylic acid,



and 2-amino-1-methylcyclohexane-2-carboxylic acid. The first acid is obtained by treating 1-methylcyclohexan-4-one (Wallach, Abstr., 1906, i, 514) with concentrated aqueous potassium cyanide and ammonium chloride, and hydrolysing the resulting amino-nitrile (*hydrochloride*, $\text{C}_8\text{H}_{14}\text{N}_2\text{HCl}$, m. p. 191° , decomp.) by ten hours' boiling with concentrated hydrochloric acid; it has m. p. above 300° , forms a blue copper salt, $(\text{C}_8\text{H}_{14}\text{O}_2\text{N})_2\text{Cu} \cdot \text{H}_2\text{O}$, has a neutral reaction, and is tasteless. Nitrous acid converts it into the corresponding *hydroxy-acid*, $\text{C}_8\text{H}_{14}\text{O}_3$, m. p. 115° , whilst the reaction between equal molecular quantities of the acid and phenylcarbimide in alkaline solution leads to the formation of the



m. p. 190° , which, warmed with 25% hydrochloric acid, yields the corresponding cyclic *hydantoin*, $\text{C}_{15}\text{H}_{18}\text{O}_2\text{N}_2$, m. p. 184° .

2-Amino-1-methylcyclohexane-2-carboxylic acid is best obtained by heating 1-methylcyclohexan-2-one, dissolved in light petroleum, for two hours at 60° with potassium cyanide, ammonium chloride, and a little water, and hydrolysing the resulting amino-nitrile (the *hydrochloride*, $\text{C}_8\text{H}_{14}\text{N}_2\text{HCl}$, decomposes at 182°) by ten hours' heating with concentrated hydrochloric at 100° under pressure; 1-methylcyclohexan-2-one is obtained as a by-product. The acid has a sweet taste, and melts above 300° ; the *naphthylcarbamido-acid*, $\text{C}_{19}\text{H}_{22}\text{O}_2\text{N}_2$, has m. p. 180° (decomp.). In this acid, steric hindrance caused by the neighbouring methyl group (compare also Skita, *loc. cit.*) is observed, not only during the hydrolysis of the preceding amino-nitrile, but also in the esterification of the acid and in the hydrolysis of the resulting ester. Whilst 4-amino-1-methylcyclohexane-4-carboxylic acid is readily esterified by alcoholic hydrogen chloride, the isomeric acid must be saturated in alcoholic solution with hydrogen chloride and heated for ten hours at 100° under pressure. The esters are very reactive liquids, with a basic odour and an alkaline reaction.

Ethyl 4-amino-1-methylcyclohexane-4-carboxylate, $\text{C}_{10}\text{H}_{19}\text{O}_2\text{N}$, b. p. $103-105^\circ/11$ mm., $D_{20}^{20} 0.928$, is easily hydrolysed by boiling water, produces local anaesthesia like the esters of δ -amino-acids, and forms a *picrate*, $\text{C}_{16}\text{H}_{25}\text{O}_3\text{N}$, m. p. 185° , and a *phenylthiocarbamide*, $\text{C}_{17}\text{H}_{21}\text{O}_2\text{N}_2\text{S}$, m. p. $205-206^\circ$. By treating the ester in chloroform with *D*-alanine chloride and hydrolysing the resulting ester, a mixed aliphatic, alicyclic dipeptide is obtained, $\text{C}_{11}\text{H}_{20}\text{O}_2\text{N}_2$, which has m. p. above 300° , and $[\alpha]_D^{20} 22.33^\circ$ in 21% hydrochloric acid. *Ethyl 2-amino-1-methylcyclohexane-2-carboxylate*, b. p. $99-100^\circ/12$ mm., forms a *phenylthiocarbamide*, m. p. $198-199^\circ$, and requires heating with sodium hydroxide for hydrolysis to occur.

The esters of both acids are converted by twenty to twenty-four hours' heating at 200° into cyclic derivatives of 2:5-diketopiperazine,

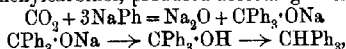
$C_7H_{12} \begin{smallmatrix} \text{CO}\cdot\text{NH} \\ \text{NH}\cdot\text{CO} \end{smallmatrix} C_7H_{12}$, which melt above 300° ; the one derived from 4-amino-1-methylcyclohexane-4-carboxylic acid can be partly hydrolysed by concentrated hydrobromic acid at 100° under pressure, yielding a cyclic dipeptide, $CO_2H\cdot C_7H_{12}\cdot NH\cdot CO\cdot C_7H_{12}\cdot NH_2$, which is isolated as the reddish-violet copper salt, $(C_{10}H_{27}O_5N_{2\frac{1}{2}}Cu$.

C. S.

New Synthesis of Aromatic Carboxylic Acids from the Hydrocarbons. PAUL SCHORIGIN (*Ber.*, 1908, 41, 2723—2728).—The author has shown that when carbon dioxide acts on a mixture of sodium and mercury alkyl in ethereal solution, the corresponding aliphatic acid is produced (this vol., i, 882). When dry benzene is used instead of ether in this reaction, in addition to the aliphatic acid, benzoic acid is formed in considerable amount. The reaction is due, as in the previous case, to the intermediate formation of sodium alkyl, for by the interaction of carbon dioxide, sodium, and benzene, or of carbon dioxide, mercury diethyl, and benzene, benzoic acid is not obtained. In this reaction the mercury diethyl can be replaced by zinc diethyl, but the yields are smaller: $C_6H_6 + C_2H_5Na = C_6H_5Na + C_2H_5$; $C_6H_5Na + CO_2 = C_6H_5\cdot CO_2Na$.

The reaction proceeds also in the case of other aromatic hydrocarbons, but the carboxyl group enters the side-chain; thus toluene gives phenylacetic acid, *m*-xylene gives *m*-tolylacetic acid, and ethylbenzene yields α -phenylpropionic acid. When zinc diethyl is used instead of mercury diethyl, toluene gives *p*-toluic acid in addition to phenylacetic acid.

In addition to these acids, neutral by-products are sometimes formed; thus benzene yields a small amount of triphenylmethane mixed with triphenylcarbinol, produced according to the scheme:



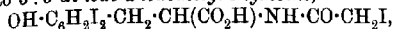
and ethylbenzene gives a hydrocarbon, $C_{15}H_{18}$, which is a colourless liquid with a weak, sweetish, aromatic odour, b. p. $161\text{--}163^\circ/13$ mm., D_4^{20} 0.9858, D_4^{25} 0.9685, and is very stable towards oxidising agents. Negative results were obtained on applying the reaction to cyclohexane and diisomyl.

J. C. C.

Derivatives of 3:5-Di-iodo-*L*-tyrosine. EMIL ABDERHALDEN and MARKUS GUGGENHEIM (*Ber.*, 1908, 41, 2852—2857. Compare this vol., i, 420).—Acid chlorides containing iodine, when coupled with amino-acids, yield compounds which crystallise better than the corresponding bromine derivatives. Their use is attended, however, with the disadvantage that they cause racemisation; thus, *d*-bromopropionic acid and potassium iodide react to give optically inactive iodopropionic acid.

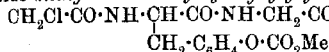
Iodoacetic acid is obtained in colourless plates, m. p. 83° , by the interaction of potassium iodide and monochloroacetic acid. Iodoacetyl-chloride, $CH_2I\cdot CO\cdot Cl$, prepared by warming the acid with thionyl-chloride, is a heavy oil, b. p. $49\text{--}52^\circ/15$ mm., D^{25} 2.25. At the same time, iodoacetic anhydride is formed in transparent, colourless crystals, m. p. 46° .

Ethyl iodoacetyltyrosine forms rhombic, colourless, crystalline plates, m. p. 120° (corr.). By the action of iodine and sodium hydroxide, it is converted into 3:5-di-iodo- α -iodoacetyl-L-tyrosine,



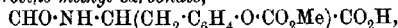
which crystallises in colourless, prismatic needles, becoming brown at 190° (corr.), and m. p. 209° (corr., decomp.). *dl*- α -Iodopropionic acid is conveniently prepared in colourless crystals, m. p. 45.5° (corr.), by the interaction of bromopropionic acid with potassium iodide. *dl*- α -Iodopropionyl chloride, $\text{CH}_3\cdot\text{CH}_2\cdot\text{I}\cdot\text{COCl}$, is a faintly-brown, pungent-smelling oil, b. p. $51-53^{\circ}/13$ mm., D_{25}^{25} 1.989. *Ethyl dl*- α -iodopropionyl-L-tyrosine crystallises in colourless plates, m. p. 126° , to a colourless oil. *dl*-3:5-Di-iodo- α -iodopropionyl-L-tyrosine crystallises in fine needles, m. p. 210.5° (corr., decomp.). *dl*-3:5-Di-iodoalanyl-L-tyrosine, obtained by the action of ammonia on the foregoing, has m. p. $217-219^{\circ}$, $[\alpha]_D^{20} + 47.23^{\circ}$. E. F. A.

The Synthesis of Polypeptides. XXV. Derivatives of Tyrosine and of Aminoacetal. EMIL FISCHER (*Ber.*, 1908, 41, 2860-2875).—In part already published (this vol., i, 544). A tetrapeptide containing tyrosine was obtained by the following method. The hydrochloride of *glycyl-d*-alanine methyl ester, prepared by the action of gaseous hydrogen chloride and dry methyl alcohol on the dipeptide, forms thin, colourless, silky, glistening prisms, m. p. $160-162^{\circ}$ (corr.). This salt is decomposed by a little less than the calculated quantity of sodium methoxide, and is immediately used for further synthesis, as it easily passes over into glycine-*d*-alanine anhydride. Coupled with chloroacetyltyrosyl chloride methyl carbonate, it yields *methyl chloroacetyl-tyrosyl-glycyl-d*-alanine ester methyl carbonate,



This ester crystallises in concentrically-arranged, short prisms of a faint yellow colour, which sinter at 200° , m. p. 208° (corr.), to a yellow liquid. On hydrolysis, the tetrapeptide, *glycyl-tyrosyl-glycyl-d*-alanine, is formed. This is an amorphous, faintly yellow powder, which, quickly heated, colours at $180-190^{\circ}$ and decomposes at 225° (corr.). It is precipitated like the albumoses by saturated ammonium sulphate in concentrated solution, but not in dilute solution. It is probably a mixture of stereoisomerides.

Formyl-L-tyrosine methyl carbonate,

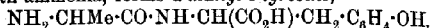


crystallises from water in glistening, large, thin plates, which sinter at 142° , m. p. 147° (corr.), $[\alpha]_D^{20} + 66.7^{\circ}$. *Formyl-L*-tyrosine ethyl carbonate forms centimetre-long, thin, silky, glistening needles, m. p. $177-179^{\circ}$ (corr., decomp.), after sintering at 173° . E. F. A.

Synthesis of Polypeptides. XXVIII. Derivatives of Glycine, *d*-Alanine, *l*-Leucine, and *l*-Tyrosine. EMIL ABDEHARDEN and ALFRED HIRSZOWSKI (*Ber.*, 1908, 41, 2840-2851).—The presence of *l*-tyrosine in polypeptides may possibly give them properties similar to those of the albumoses, but, in addition, the position of the tyrosine

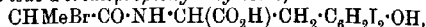
in the molecule must be defined. The polypeptide isolated by Fischer and Abderhalden (Abstr., 1907, i, 737, 990) from silk consists of two molecules of glycine, one of *d*-alanine, and one of *l*-tyrosine; on partial hydrolysis, it yields glycyl-*d*-alanine anhydride and glycyl-*l*-tyrosine anhydride. Of the eight possible stereoisomeric polypeptides of this composition, but one has been prepared synthetically; this, however, is not precipitated by ammonium sulphate. The polypeptides now described containing tyrosine are likewise not precipitated by ammonium sulphate.

Ethyl *l*-tyrosine and *d*- α -bromopropionyl chloride were coupled by Fischer's method to ethyl *d*- α -bromopropionyl-*l*-tyrosine, which crystallises in bunches of long plates, m. p. 133.5—134.5° (corr.). On hydrolysis, it is converted into *d*- α -bromopropionyl-*l*-tyrosine, which forms crystalline plates pointed at both ends, m. p. 165° (corr.), and, when treated with ammonia, forms *d*-alanyl-*l*-tyrosine,

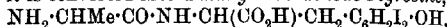


The dipeptide crystallises in large, hexagonal plates, m. p. (frothing) 202° (decomp. at 285°, corr.), and has $[\alpha]_D^{20} + 43.14^\circ$. It is rapidly hydrolysed by the pancreas extract of the dog.

d-3:5-Di-iodo- α -bromopropionyl-*l*-tyrosine,



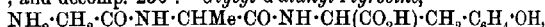
prepared by the action of iodine in chloroform on bromopropionyl-tyrosine ester dissolved in sodium hydroxide, crystallises in rosettes of long, thin needles, m. p. 217° (corr., decomp.). By the action of ammonia, it is converted into *d*-alanyl-3:5-di-iodo-*l*-tyrosine,



which, on heating, becomes yellow at 188°, brown at 205°, and has m. p. 231° (corr., decomp.).

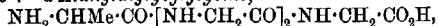
d- α -Bromoisohexoyl-*l*-tyrosine is obtained as an amorphous solid, m. p. 141.5° (corr.), by the interaction of ethyl tyrosine and *d*- α -bromoisohexoyl chloride. *l*-Leucyl-*l*-tyrosine, as prepared by the action of aqueous ammonia on the foregoing, is amorphous. It has m. p. 269° (corr., decomp.), $[\alpha]_D^{20} + 10.37^\circ$, and is hydrolysed by pancreas extract.

Chloroacetyl-*d*-alanyl-*l*-tyrosine, prepared by coupling chloroacetyl chloride with *d*-alanyl-*l*-tyrosine, is amorphous, sinters at 97°, m. p. 108°, and decomp. 236°. Glycyl-*d*-alanyl-*l*-tyrosine,



is also amorphous; it becomes yellow at 193°, m. p. 208° (corr., decomp.), and has $[\alpha]_D^{20} - 4.83^\circ$. It gives an amorphous precipitate with phosphotungstic acid, but only a slight clouding with ammonium sulphate.

d- α -Bromopropionyl-diglycylglycine, prepared from diglycylglycine and *d*-bromopropionyl chloride, crystallises in bunches of slender needles, m. p. 189.5°. *d*-Alanyldiglycylglycine,

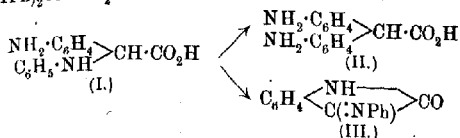
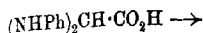


crystallises in minute needles, m. p. 253.7° (corr., decomp.), $[\alpha]_D^{20} + 37^\circ$, and is rapidly attacked by pancreas juice.

E. F. A.

Action of Dichloroacetic Acid on Aniline and its Homologues. II. IWAN OSTROMISLENSKY (*Ber.*, 1908, 41, 3019—3023. Compare this vol., i, 82).—In the direct action of dichloroacetic acid

with an excess of an arylamine, the various stages appear to be those represented by the following formulæ :



The dianilinoacetic acid is unstable, and undergoes the Hofmann rearrangement either singly or in duplicate. In the former case, the acid formed (I) loses water and yields imesatin (III), and in the latter case the product is 4:4'-diaminodiphenylacetic acid (II). The dianilinoacetic acid cannot be isolated from the products of the reaction on account of its instability in the presence of an excess of the amine; it may, however, be obtained by the action of aniline on glyoxylic acid.

4:4'-Diaminodiphenylacetic acid crystallises from alcohol, in which imesatin is readily soluble, as soft, dark-coloured flakes, which, when powdered, have a pale canary-yellow colour. It melts at 73–74° to a dark brown oil, resolidifies, and then has m. p. 233–234° (decomp.). The crystals contain 1 mol. of alcohol, which they lose when heated for fifteen hours at 90°. The product is then dark yellow, and decomposes at 215°. The presence of amino-groups is shown by the readiness with which it can yield azo-dyes, and its constitution follows (a) from the fact that, when heated with dilute hydrochloric acid, it loses carbon dioxide and yields 4:4'-diaminodiphenylmethane, and (b) from the presence of four active hydrogen atoms (compare Zerewitinoff, this vol., i, 593).

A second modification (β) of the diamino-acid is obtained when ether is added to its alcoholic solution. It is excessively unstable, and, when brought into contact with the air, yields a dark-coloured oil, which is transformed into the original modification (a) when kept over sulphuric acid.

J. J. S.

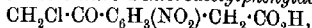
Action of Glyoxylic Acid and of Diacetyl-glyoxylic Acid on Aniline and its Homologues. IWAN OSTROMISLENSKY (*Ber.*, 1908, 41, 3029–3034).—The product described by Böttlinger (*Abstr.*, 1879, 51) as the aniline salt of anilglyoxylic acid is shown to be the unstable dianilinoacetic acid. This follows (a) from the readiness with which aniline is eliminated, yielding anilglyoxylic acid, and (b) from the readiness with which it is transformed into 4:4'-diaminodiphenylacetic acid.

Di-p-toluidinoacetic acid, obtained from glyoxylic acid and *p*-toluidine, forms hard, colourless crystals, m. p. about 96°, becoming yellowish-red when kept. *Ethyl di-p-toluidinoacetate*, $\text{CH}(\text{NH}\cdot\text{C}_6\text{H}_4\text{Me})_2\cdot\text{CO}_2\text{Et}$, obtained from the base and ethyl glyoxylate, forms colourless crystals, m. p. 170°, and readily yields *p*-tolyl-*p*-methylimesatin when warmed with mineral acids.

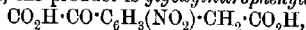
J. J. S.

The Three Chloroacetyl-benzyl Cyanides [Chloroacetyl-phenylacetoneitriles] and Their Derivatives. FRANZ KUNCHELL [and F. Flos] (*Ber.*, 1908, 41, 3046—3050. Compare Abstr., 1906, i, 848).—Chloroacetyl chloride reacts much more vigorously with phenylacetoneitrile in the presence of aluminium chloride than with acetyl chloride. When the condensation is carried out in the presence of carbon disulphide, the product consists of a mixture of *o*-, *m*-, and *p*-chloroacetylphenylacetoneitriles. The para-compound solidifies when the crude product is kept, and is freed from the two isomerides by pressing on porous plate. It crystallises from a mixture of benzene and light petroleum in colourless needles, m. p. 93°. The ortho-compound is obtained as a solid when the oily residue from the para-compound is distilled under diminished pressure and kept for some days, or a large yield can be obtained from the acid liquor formed when the initial condensation product is added to water. It crystallises from water in glistening, snow-white needles, m. p. 47°. *m*-Chloroacetylphenylacetoneitrile is an oil, b. p. 210—215°/25 mm. Each of the three compounds can be oxidised by alkaline hydrogen peroxide to the corresponding phthalic acid.

p-Chloroacetylphenylacetic acid, $\text{CH}_2\text{Cl}\cdot\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, prepared by hydrolysing the nitrile with fuming hydrochloric acid at 120—130°, forms yellow needles, m. p. 130°. Fuming nitric acid converts the nitrile into 3-nitro-4-chloroacetylphenylacetic acid,



which crystallises from water in yellow, glistening needles, m. p. 149°. Its solutions in alkalis have a deep blue colour, but turn yellow when boiled. When the nitrile is heated for two hours at 70—100° with fuming nitric acid, the product is glycoylnitrophenylacetic acid,



which crystallises from ether in yellow needles, m. p. 214°. Its solutions in alkali hydroxides are blue, but in ammonia, yellow.

J. J. S.

A Dextrorotatory Resin Acid. W. SCHKATELOFF (*Chem. Zentr.*, 1908, ii, 807; from *Mon. Sci.*, 1908, 22, [ii], 548).—If a solution of crude β -sylvic acid, saturated with sulphur dioxide, is allowed to crystallise, the mother liquor will, after a time, deposit *d*-sylvic acid as a crystalline powder. When recrystallised from ether, it is obtained in rectangular plates, soluble in alkaline solutions, $[\alpha]_D + 78^\circ$.

J. V. E.

Esters of Hexahydrobenzoylacetic Acid. ANDRÉ WAHL and A. MEYER (*Bull. Soc. chim.*, 1908, [iv], 3, 957—963).—Most of the results recorded have been published already (Abstr., 1907, i, 765), but detailed accounts of the methods of preparation and new constants for the various substances described are now given.

Ethyl hexahydrobenzoylacetate, $\text{C}_6\text{H}_{11}\cdot\text{CO}\cdot\text{CH}_2\cdot\text{CO}_2\text{Et}$, b. p. 145—147°/19 mm., $D^{20} 1.039$, $D^{18} 1.025$ (compare Zelinsky and Schwedoff, Abstr., 1907, i, 704), is a colourless liquid, insoluble in water, soluble in most organic solvents, and gives a violet coloration with ferric chloride; the copper salt, m. p. 120°, separates from a

mixture of ether and light petroleum in green leaflets. The methyl ester, b. p. $138^{\circ}/20$ mm., yields a similar copper salt, m. p. $137-138^{\circ}$. With phenylhydrazine, the ethyl ester furnishes 1-phenyl-3-cyclohexyl-5-pyrazolone, $\text{NPh} \begin{array}{c} \text{N}=\text{C} \cdot \text{C}_6\text{H}_{11} \\ \text{CO} \cdot \text{OH}_2 \end{array}$, m. p. 126° , which crystallises in colourless spangles from boiling alcohol. With hydroxylamine, 3-cyclohexylisooxazolone, $\text{C}_6\text{H}_{11} \cdot \text{C} \begin{array}{c} \text{CH}_2 \cdot \text{CO} \\ \text{N} \text{---} \text{O} \end{array}$, m. p. 42° , is produced. This crystallises in needles, several centimetres in length, and is readily soluble in alcohol or ether, less so in water or light petroleum.

The residues obtained in the preparation of ethyl hexahydrobenzoyl-acetate deposit eventually slender, silky, colourless needles of a substance, m. p. $90-91^{\circ}$, which is probably 3-hexahydrobenzoyl-6-cyclohexyl-2:4-pyrrone, $\text{C}_6\text{H}_{11} \cdot \text{C} \begin{array}{c} \text{CH} \cdot \text{CO} \\ \text{O} \text{---} \text{CO} \text{---} \text{CH} \cdot \text{CO} \cdot \text{C}_6\text{H}_{11} \end{array}$ T. A. H.

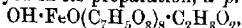
Salicylic Acid-Iron Reaction. KARL HOPFGARTNER (*Monatsh.* 1908, 29, 689—712).—An account is given of the views of various authors as to the cause of the violet coloration produced by adding salicylic acid to solutions of ferric salts. A new study of the reaction has led to the isolation of two *ferrisalicylic acids*:

(I) $\text{OH} \cdot \text{Fe}(\text{C}_7\text{H}_5\text{O}_3)_2, \text{H}_2\text{O}$ and (II) $\text{OH} \cdot \text{Fe}_2\text{O}(\text{C}_7\text{H}_5\text{O}_3)_6$.

The compound (I), which is obtained when slightly less than 3 mols. of sodium salicylate are added to 1 mol. of ferric chloride in moderately concentrated aqueous solution at the ordinary temperature, or on prolonged shaking of ferric hydroxide with salicylic acid and water, forms blackish-violet, microscopic needles, is stable at 100° , but decomposes at higher temperatures, and dissolves in water or dilute ferric chloride, forming a violet solution; hence it is to be considered as the cause of the violet coloration of the salicylic acid-iron reaction. The compound (II), formed by the action of sodium salicylate on ferric chloride, in hot concentrated solution, or by heating the acid (I) with salicylic acid at $155-160^{\circ}$, crystallises from ether-alcohol in scarlet, microscopic prisms containing ether, becomes blackish-brown when dried at 100° , decomposes at higher temperatures, and is converted into the acid (I) on prolonged shaking with water, the conversion being accelerated by gentle heating.

Ferric chloride and sodium 5-bromosalicylate form, in the same manner, two *ferribromosalicylic acids*, $\text{OH} \cdot \text{Fe}(\text{C}_7\text{H}_4\text{O}_3\text{Br})_2, \frac{1}{2}\text{H}_2\text{O}$ and $\text{OH} \cdot \text{Fe}_2\text{O}(\text{C}_7\text{H}_4\text{O}_3\text{Br})_6$; *p*-hydroxybenzoic acid, on the other hand, is found to form only one *ferric*-compound, $\text{OH} \cdot \text{Fe}(\text{C}_7\text{H}_5\text{O}_3)_2, \text{H}_2\text{O}$.

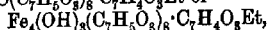
Hantzsch and Desch's ferrisalicylic acid (*Abstr.*, 1902, i, 708), prepared from ferric chloride, sodium acetate, and salicylic acid, is considered to have the constitution $\text{Fe}_4(\text{OH})_3(\text{C}_7\text{H}_5\text{O}_3)_9$, and to be related, therefore, to the ferrisalicylic acid (I). If insufficient salicylic acid is employed in its preparation, a *product*,



is obtained.

In agreement with Hantzsch and Desch's view, that in such

compounds the ferric atom displaces phenolic hydrogen atoms, it is found that, when heated with alcohol, ferrisalicic acid (II) forms an *ethyl ester*, $\text{OH} \cdot \text{Fe}_3\text{O}(\text{C}_7\text{H}_5\text{O}_2)_3 \cdot \text{C}_2\text{H}_5\text{O}_2\text{Et}$ or



which points to the presence of a free carboxyl in the original compound. A similar *ethyl ester*, $\text{OH} \cdot \text{Fe}_3\text{O}(\text{C}_7\text{H}_4\text{O}_2\text{Br})_3 \cdot \text{C}_2\text{H}_5\text{O}_2\text{BrEt}$, is obtained from the second ferribromosalicylic acid. Moreover, a crystalline *ferric*-compound, $\text{Fe}(\text{C}_7\text{H}_5\text{O}_2)_3$, is formed by the action of salicylaldehyde and 10% potassium hydroxide on ferric chloride in concentrated solution. On the other hand, the ferrisalicic acid (II) is of complex nature, since it does not form silver salicylate with silver nitrate in alcoholic solution, a reaction which takes place directly with sodium salicylate; the salicylic acid in the complex is considered to be "masked" in the same manner as is the sulphuric acid in green chromium sulphate. G. Y.

Methyl-carbonato-derivatives of Phenolcarboxylic Acids and their Use for Synthetical Operations. EMIL FISCHER (*Ber.*, 1908, 41, 2875—2891).—Chlorides of phenolcarboxylic acids cannot be prepared by ordinary methods, as the chlorides of phosphorus attack both carbonyl and hydroxyl, and yield products containing phosphorus. When, however, by means of methyl chlorocarbonate, the phenol group is first converted into a methyl-carbonato-compound, a normal acid chloride results, which is available for manifold synthetical purposes. On hydrolysis of the new products with cold alkali hydroxide, the original phenolic group is regenerated (compare this vol., i, 544).

p-Methyl-carbonatobenzoic acid, $\text{CO}_2\text{Me} \cdot \text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{CO}_2\text{H}$, is obtained almost quantitatively from *p*-hydroxybenzoic acid and methyl chlorocarbonate. It crystallises in colourless, short needles, m. p. 179° (corr.), and gives no coloration with ferric chloride or with Millon's reagent. *p*-Methyl-carbonatobenzoyl chloride, prepared by the action of phosphorus pentachloride on the above, forms colourless, long needles, m. p. 82—83° (corr.), and distils without decomposition by 10—15 mm.

It combines with glycine ester to *ethyl p*-methyl-carbonatobenzoyl-glycine, $\text{CO}_2\text{Me} \cdot \text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{CO} \cdot \text{NH} \cdot \text{CH}_2 \cdot \text{CO}_2\text{Et}$, which crystallises in needles, m. p. 63° (corr.), and, when hydrolysed with sodium hydroxide, yields *p*-hydroxyhippuric acid, $\text{OH} \cdot \text{C}_6\text{H}_4 \cdot \text{CO} \cdot \text{NH} \cdot \text{CH}_2 \cdot \text{CO}_2\text{H}$. This separates in colourless, thin, glistening prisms, m. p. 240° (corr.), to a yellow liquid, and subsequently decomposes; it gives a brown coloration with ferric chloride, and is coloured red by Millon's reagent. It is, perhaps, identical with an acid, m. p. 228°, isolated from pathological urine.

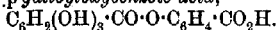
3 : 4-Dimethyl-carbonatobenzoic acid, $(\text{CO}_2\text{Me} \cdot \text{O})_2\text{C}_6\text{H}_3 \cdot \text{CO}_2\text{H}$, is obtained in colourless needles, m. p. 165—166°. 3 : 4-Dimethyl-carbonatobenzoyl chloride crystallises in bunches of needles, m. p. 118° (corr.), which sinter 2° or 3° lower.

3 : 4 : 5-Trimethyl-carbonatobenzoic acid, $(\text{CO}_2\text{Me} \cdot \text{O})_3\text{C}_6\text{H}_2 \cdot \text{CO}_2\text{H}$, is obtained by the interaction of gallic acid and methyl chlorocarbonate in an atmosphere of hydrogen in a special apparatus. It forms colourless, thin prisms, which sinter about 130°, m. p. 136—141°. It forms a

crystalline, colourless *bromo-derivative* and a soluble *pyridine* salt, which is decomposed by excess of the base and liberates carbon dioxide. It is completely hydrolysed by acids, alkaline hydroxides, or ammonia to gallic acid; the methylcarbonato-group is eliminated as methyl carbamate by ammonia. Cautious partial hydrolysis in an atmosphere of hydrogen yields *hydroxydimethylcarbonatobenzoic acid*, $(\text{CO}_2\text{Me}\cdot\text{O})_2\cdot\text{C}_6\text{H}_3(\text{OH})\cdot\text{CO}_2\text{H}$. This has m. p. 160° (corr.), and gives a dark green coloration with ferric chloride.

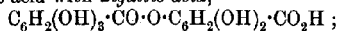
3:4:5-*Trimethylcarbonatobenzoic acid* crystallises in colourless, glistening plates, m. p. $96-97^\circ$ (corr.).

3:4:5-*Trimethylcarbonatobenzoyl chloride* forms long needles, m. p. 86° (corr.) to a clear liquid; it reacts with dimethylaniline and also with aniline, forming in the latter case *trimethylcarbonatobenzoic anilide*, which crystallises in needles pointed at both ends, m. p. $175-176^\circ$ (corr.) to a clear liquid. *p*-*Trimethylcarbonatobenzoyloxybenzoic acid*, $(\text{CO}_2\text{Me}\cdot\text{O})_3\cdot\text{C}_6\text{H}_2\cdot\text{CO}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$, is obtained as an oil, which solidifies to thin prisms aggregated in bunches, m. p. 165° (corr.). On hydrolysis, for which purpose the crude product may be used with advantage, it yields *p*-*galloyloxybenzoic acid*,



This is obtained as a sandy, crystalline powder, consisting of obliquely cut plates, m. p. 260° (corr., decomp.). It gives similar colorations to gallic acid with ferric chloride or potassium cyanide.

On coupling trimethylcarbonatobenzoyl chloride with hydroxydimethylcarbonatobenzoic acid in alkaline solution, an amorphous mass was obtained, which contained much *pentamethylcarbonatobenzoyloxybenzoic acid*, and, on hydrolysis, yielded what appeared to be a mixture of gallic acid with *digallic acid*,



it formed short prisms or needles, m. p. $275-280^\circ$ (decomp.).

E. F. A.

Nitration. IV. Nitration of *N*-Acyl Compounds of Aniline derived from Certain Polybasic, Aliphatic, and Aromatic Acids. J. BISHOP TINGLE and F. C. BLANCK (*J. Amer. Chem. Soc.*, 1908, 30, 1587-1599. Compare this vol., i, 778).—A study has been made of the nitration of anilides of various di- and tri-basic acids, the experiments being carried out under the same general conditions as those described previously.

Oxanilic acid yields the *p*-nitro-derivative, both on direct nitration and also on nitration in presence of oxalic acid. Oxanilide gives the *m*-nitro-derivative in presence of sulphuric acid, and the *p*-dinitro-derivative in presence of acetic acid. Succinanilic acid yields the *m*-nitro-derivative in presence of sulphuric acid, and the *p*-nitro-derivative in presence of oxalic acid. By direct nitration of succinanil, a mixture of ortho- and para-derivatives is obtained, but in presence of sulphuric or oxalic acid, the *p*-nitro-derivative is formed. On direct nitration, succinanilide yields the *s*-*p*-dinitro-derivative, and tartranilide gives the *p*-nitro-derivative.

Phthalanil, both on direct nitration and in presence of oxalic acid,

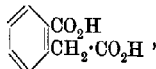
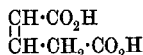
yields the *p*-nitro-derivative. Citranilide, on direct nitration, gives a trinitro-derivative.

The experiments so far carried out have led to the following generalisations. Direct nitration yields either the para-derivative or a mixture of ortho- and para-derivatives, except in the case of benzanilide, which gives some meta-derivative. Nitration in presence of sulphuric acid gives chiefly, or exclusively, the *m*-nitro-derivative. Oxalic acid does not affect the course of the nitration, and this is probably due to its small solubility in nitric acid.

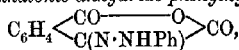
The possible modes of representing the disubstituted benzenes are discussed, and it is stated that the authors' results are fully and satisfactorily accounted for by Barlow and Pope's formula for benzene (*Trans.*, 1906, 89, 1697).

A discussion is also given of the mechanism of substitution, and leads to the conclusion that substitution must be regarded as essentially an additive phenomenon (compare Armstrong, *Proc.*, 1891, 7, 89). E. G.

Homophthalic Esters, Oxymethylenehomophthalic Esters, and their *iso*Coumarin and *iso*Carbostyryl Derivatives. WALTHER DIECKMANN and WILHELM MEISER (*Ber.*, 1908, 41, 3253—3269).—The structural analogy existing between homophthalic and glutaconic acids:

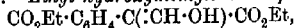


suggested an investigation into the properties of homophthalic acid with a view to comparing them with those of the other acid. The methylene group, which is very reactive in glutaconic acid (compare Henrich, *Abstr.*, 1902, i, 422), is markedly less reactive in the aromatic derivative. Sodium ethoxide and benzyl bromide are without action on ethyl homophthalate, as is also diazobenzene chloride. With homophthalic anhydride, however, the diazo-salt forms *benzeneazohomophthalic anhydride* [*phthalonic anhydride phenylhydrazones*],



which crystallises from benzene in long, reddish-yellow needles, m. p. 199°. It is hydrolysed by acids or alkalis to 3-phenylphthalazone-1-carboxylic acid. This marked difference between the acid and its anhydride is ascribed to the formation of the ring. Ethyl homophthalate yields naphthalene derivatives on condensation with sodium ethoxide (compare Pechmann, Bauer, and Obermiller, *Abstr.*, 1904, i, 592).

A comparison of the hydroxymethylene derivatives of the two acids shows a strong resemblance between them (compare Pechmann, *Abstr.*, 1893, i, 401), but the condensation proceeds more easily with the benzene compound. *Ethyl hydroxymethylenehomophthalate*,

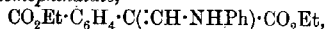


obtained in good yield from ethyl formate, ethyl homophthalate, and sodium as a colourless, viscid oil, is converted by heating at 100° in two hours into *ethyl isocoumarin-4-carboxylate*, $\text{C}_6\text{H}_4 \begin{array}{c} \text{CO} \text{---} \text{O} \\ \diagdown \quad \diagup \\ \text{C}(\text{CO}_2\text{Et}) \end{array} \text{CH}_2$,

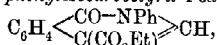
which crystallises in colourless needles from alcohol, m. p. 67—68°. Methyl-alcoholic potassium hydroxide hydrolyses it to formic and homophthalic acids. The corresponding methyl ester, $C_{11}H_8O_4$, forms colourless needles, m. p. 97°. *isocoumarin-4-carboxylic acid*, $C_{10}H_6O_4$, obtained by warming the ester with concentrated hydrochloric acid, forms slender needles, m. p. 244°, and yields isocoumarin on heating with 50% sulphuric acid. This acid is similar to the corresponding 3-carboxylic acid (Bamberger and Frew, Abstr., 1894, i, 192), but its ready conversion into formic and homophthalic acids by alkali is in marked contrast with the isomeric acid.

Ethyl isocarbostyryl-4-carboxylate, $C_6H_4 \begin{smallmatrix} \text{CO-NH} \\ \text{C(CO}_2\text{Et)} \end{smallmatrix} \text{CH}_3$, prepared either from ethyl hydroxymethylenehomophthalate or from ethyl isocoumarincarboxylate by the action of ammonia, crystallises in colourless, slender needles, m. p. 227°. By shaking isocoumarin-4-carboxylic acid with ammonia and acidifying, isocarbostyryl-4-carboxylic acid, $C_{10}H_7O_4N$, is obtained as needles from glacial acetic acid, m. p. 290° (decomp.). If, however, the ammonia is warmed, isocarbostyryl is precipitated. *Ethyl 2-methylisocarbostyryl-4-carboxylate*, $C_6H_4 \begin{smallmatrix} \text{CO-NMe} \\ \text{C(CO}_2\text{Et)} \end{smallmatrix} \text{CH}_3$, obtained from methylamine, crystallises from methyl alcohol in glistening needles, m. p. 98°. The corresponding acid, $C_{11}H_9O_4N$, forms small needles, m. p. 262°.

Aniline and ethyl hydroxymethylenehomophthalate form *ethyl anilinoethylenehomophthalate*,



which crystallises in needles, m. p. 102.5°. By heating them together for a long time, *ethyl 2-phenylisocarbostyryl-4-carboxylate*,



is produced; it forms crystals, m. p. 118°. The parent acid, $C_{16}H_{11}O_4N$, forms microscopic needles, m. p. 267°.

Experiments were carried out in the hope that stereoisomerides of hydroxymethylenehomophthalic ester would exist, but only with partial success. By the action of *m*-nitrobenzoyl chloride on the sodium salt of methyl hydroxymethylenehomophthalate, two isomeric *m*-nitrobenzoates were obtained. The α -compound, $C_{19}H_{15}O_6N$, obtained when the dry sodium salt in ether is treated with *m*-nitrobenzoyl chloride, crystallises from methyl alcohol in prisms, m. p. 139°. The β -compound, formed when the aqueous solution is treated with the chloride, crystallises in slender needles, m. p. 98°, but experiments to transform the one isomeride into the other were without the desired result.

When ethyl hydroxymethylenehomophthalate is coupled with diazobenzene chloride, ring formation takes place, *ethyl 3-phenylphthalazone-1-carboxylate*, $C_6H_4 \begin{smallmatrix} \text{CO-NPh} \\ \text{C(CO}_2\text{Et)} \end{smallmatrix} \text{N}$, being produced, which forms prisms, m. p. 115°.

W. R.

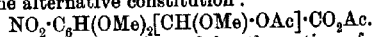
Methylenedioxyhydratropic Acid. Tiffeneau's Migration of the Phenyl Group. PAUL HÖRNING (*Ber.*, 1908, 41, 3081—3084. Compare this vol., i, 497).—From the investigations of Tiffeneau, it

was to be expected that derivatives of hydratropic acid could be prepared from α -ethoxy- α -phenylethylene, α -ethoxyanethole, and α -ethoxyisosafole by acting on them with mercuric oxide and iodine in alcoholic solution and treating the ethoxyiodohydrins so formed with alcoholic potassium hydroxide. The experiments carried out with the first two substances were unsuccessful, but methylenedioxyhydratropic acid was prepared from isosafole.

α -Methoxyisosafole is converted by mercuric oxide and iodine in methyl alcohol into the *iodohydrin*, $\text{CH}_3\text{O}_2\text{C}_6\text{H}_4\text{C}(\text{OMe})_2\text{CHMeI}$, which, however, could not be isolated, since it readily parts with iodine. The iodohydrin is converted by an alcoholic solution of potassium hydroxide into methylenedioxyhydratropic acid, $\text{C}_{10}\text{H}_{10}\text{O}_4$, m. p. $78.5-79^\circ$, identical with the acid obtained by Bougault from methylenedioxyhydratropaldehyde (Abstr., 1900, i, 495). α -Ethoxyisosafole, when similarly treated, yields the same acid. W. H. G.

Nitration of Opianic Esters and Derivatives of Nitro-opiatic Acid. RUDOLF WEGSCHEIDER, NOE L. MÜLLER, and EDUARD CHIARI (*Monatsh.*, 1908, **29**, 713-747).—Whilst ψ -methyl nitro-opianate, which is remarkable in having a m. p. $(181.5-182.5^\circ)$; Abstr., 1904, i, 59) higher than that of the corresponding acid, is readily prepared, the methyl ester is formed only with difficulty, and only one ethyl ester, of uncertain constitution, has as yet been obtained. It was therefore of interest to study the formation of nitro-opianates by the nitration of the esters of opianic acid by means of various nitrating agents. It is found that ψ -methyl nitro-opianate is obtained by nitration of either methyl opianate, the normal ester undergoing transformation during the reaction. Since the nitration of ψ -ethyl opianate leads similarly to the formation of the known ethyl nitro-opianate, this is probably the ψ -ester.

The action of acetyl nitrate on ψ -methyl opianate leads directly to the formation of ψ -methyl nitro-opianate, but on methyl opianate, to that of a *diacetate*, $\text{NO}_2\text{C}_6\text{H}(\text{OMe})_2(\text{CO}_2\text{Me})\text{CH}(\text{OAc})_2$, which separates from benzene in crystals, m. p. $159-160^\circ$, yields nitro-opiatic acid on hydrolysis, is stable towards boiling methyl alcohol, and is hydrolysed only slowly by boiling water or hot $0.1N$ potassium hydroxide; when heated with sodium methoxide or with hydrogen chloride in methyl-alcoholic solution, it is converted into ψ -methyl nitro-opianate, and, on treatment with concentrated aqueous ammonia, yields nitro-opianamide. These properties show that the diacetate cannot have the alternative constitution:



The *diacetate*, $\text{C}_{16}\text{H}_{19}\text{O}_{10}\text{N}$, formed by the action of acetyl nitrate, or of nitric acid and acetic anhydride in glacial acetic acid solution, on ethyl opianate, crystallises in colourless plates, m. p. $100-101^\circ$, and has properties resembling those of the preceding diacetate. When treated with methyl alcohol, the crude product from the action of acetyl nitrate on ethyl opianate is converted into ψ -methyl nitro-opianate.

Energetic nitration of the normal opianates by means of fuming nitric acid alone or in concentrated sulphuric acid solution leads to the

formation of 4:5(or 5:6)-dinitro-2:3-dimethoxybenzoates (Wegscheider and Strauch, this vol., i, 794). *Ethyl dinitro-2:3-dimethoxybenzoate*, $C_{11}H_{11}O_8N_2$, crystallises from alcohol in white needles, m. p. 83.5—84.5°, and, on hydrolysis, yields an acid, m. p. 176—179°.

Nitro-opianic acid is now found to have the m. p. 169—170° (corr.). The sodium salt, $C_{10}H_5O_7NNa \cdot 4H_2O$, loses $4H_2O$ at 100°. The anhydride, $C_{20}H_{10}O_{13}N_2$, is formed together with the two methyl esters by the action of diazomethane on nitro-opianic acid; it separates from glacial acetic acid in crystals, m. p. 231—233°. The action of concentrated aqueous ammonia on nitro-opianic acid at the laboratory temperature leads to the formation of a *product*, decomp. 320°, which is probably the imide, $CO \begin{array}{c} \diagup N(C_8H_7O_4) \\ \diagdown NH \end{array} C:C \begin{array}{c} \diagup N(C_8H_7O_4) \\ \diagdown NH \end{array} CO$, and may be identical with the substance described by Bistrzycki and Fink (Abstr., 1898, i, 427) as bisnitro-*m*-opindolone. G. Y.

Constitution of Tannin. N. MAXIMILIAN NIERENSTEIN (*Ber.*, 1908, 41, 3015—3019. Compare Abstr., 1905, 914; 1907, i, 331; this vol., i, 90).—When an aqueous solution of tannin is boiled with hydrogen peroxide, both ellagic acid and *pentahydroxydiphenylmethylolide-carboxylic acid* (*luteo-acid*), $\begin{array}{c} C_6H(OH)_3 \cdot CO \\ CO \cdot H \cdot C_6H(OH)_2 \cdot O \end{array}$, are formed.

The latter is deposited, when the mother liquor from the ellagic acid is concentrated, as reddish-brown needles, which can be crystallised from pyridine and acetic acid. It turns brown at 305°, and decomposes at 338—342°. It dissolves in alkali hydroxide or hydrogen carbonate solutions, and also in concentrated sulphuric acid, and, when warmed with 10% sodium carbonate solution, yields its anhydride, ellagic acid.

The carboxyl group is eliminated when a pyridine solution of the luteo-acid is mixed with concentrated hydriodic acid and exposed to daylight for five days, pentahydroxydiphenylmethylolide being obtained.

Ellagic acid can be obtained by oxidising tetra-acetyldigallide with hydrogen peroxide, the reaction consisting in the elimination of two atoms of hydrogen and the union of the two benzene nuclei.

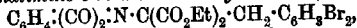
Tetra-acetyldigallide, $C_6H_3(OAc)_2 \begin{array}{c} \diagup CO \cdot O \\ \diagdown O \cdot CO \end{array} C_6H_3(OAc)_2$, crystallises from alcohol and acetic acid in needles, m. p. 130—132°.

J. J. S.

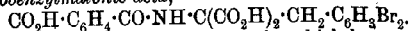
Halogen Amino-acids: 3:5-Dibromophenylalanine. HENRY L. WHEELER and SAMUEL H. CLAPP (*Amer. Chem. J.*, 1908, 40, 337—348).—The authors propose to study the halogen derivatives of phenylalanine with the view of deciding the question of the position of halogens in proteins, and in the present paper the preparation of the 3:5-dibromo-derivatives is described.

3:5-Dibromobenzyl bromide, $C_6H_3Br_2 \cdot CH_2Br$, prepared by adding bromine to 3:5-dibromotoluene at 170—180°, crystallises in prisms or long, felted needles, m. p. 95—96°, b. p. 173°/19 mm. and 169°/15 mm. It attacks the eyes violently. When heated with ethyl-sodio-

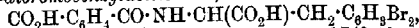
phthaliminomalonate (compare Sørensen, Abstr., 1903, i, 833), it yields *ethyl phthalimino-3:5-dibromobenzylmalonate*,



which forms aggregates of prisms and plates, m. p. 144–145°, and is hydrolysed by aqueous sodium hydroxide to *phthalimino-3:5-dibromobenzylmalonic acid*,



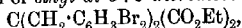
This forms snow-white, prismatic crystals, which lose carbon dioxide at 110°, or on boiling with water, with the production of *phthal. amino-3:5-dibromobenzylacetic acid*,



crystallising in clusters of minute prisms, m. p. 174° (decomp.).

When boiled with 20% hydrochloric acid, this substance is resolved into phthalic acid and 92% of the theoretical quantity of 3:5-dibromophenylalanine, $\text{CO}_2\text{H}\cdot\text{CH}(\text{NH}_2)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_3\text{Br}_2$; this dissolves in about 125 parts of water at 100°, and crystallises in long, hairy needles, m. p. 233–234° (decomp.). The aqueous solution tastes sweet and astringent. The substance is unchanged when heated with hydrochloric acid (D 1.19) in a sealed tube, but boiling aqueous barium hydroxide removes about 11% of the bromine. The *hydrochloride*, clusters of long prisms, changes on boiling with concentrated hydrochloric acid into stout prisms of a different habit, m. p. 254° (decomp.); the *barium* ($3\text{H}_2\text{O}$), *silver*, and *copper* ($1\frac{1}{2}\text{H}_2\text{O}$) salts are described; the *ethyl* ester is a viscid oil, b. p. 234–237°/24 mm., which forms a *picrate*, large, irregular prisms, m. p. 181–182°, and a *hydrochloride*, needle-like prisms, m. p. 186–187°.

The interaction of 3:5-dibromobenzyl bromide and ethylsodiummalonate leads to the formation of *ethyl di-3:5-dibromobenzylmalonate*,



crystallising in needle-like prisms, m. p. 99°.

3:5-Dibromobenzylphthalimide, $\text{C}_6\text{H}_4(\text{CO})_2\text{N}\cdot\text{CH}_2\cdot\text{C}_6\text{H}_3\text{Br}_2$, is formed by heating potassium phthalimide and 3:5-dibromobenzyl bromide to 140°. It crystallises in colourless prisms, m. p. 185°.

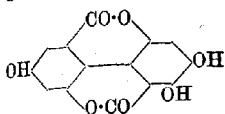
J. C. C.

Glycyrrhizic Acid. ALEXANDER TSCHIRCH and S. GAUCHMANN (*Arch. Pharm.*, 1908, 246, 545–558).—The paper discusses (1) the distribution of glycyrrhizic acid in the vegetable kingdom; (2) the best methods of preparing the acid from Russian liquorice root or the “glycyrrhizinium ammoniacale” of commerce, and (3) the nature of the hydrolytic products of the acid. It was shown previously that on hydrolysis by boiling dilute sulphuric acid, glycyrrhizic acid yields glycyrrhetic and probably glycuronic acids (Tschirch and Cederberg, Abstr., 1907, i, 545). Larger quantities of the hydrolytic products have now been prepared, and exhaustive examination shows that the second product is undoubtedly glycuronic acid.

Glycyrrhetic acid, $\text{C}_{32}\text{H}_{48}\text{O}_7$, contains one ethylene linking, possesses neither methoxyl nor ethoxyl groups, furnishes a crystalline hydrocarbon (? naphthalene) on distillation with zinc dust, an aromatic oily product when distilled with soda-lime, and, on oxidation with

permanganate, oxalic and picric acids. With nitric acid, no nitro-compound is formed, but only oxalic and picric acids. T. A. H.

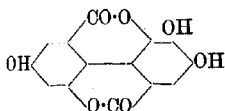
Reso flavin. V. Lactone Dyes. JOSEF HERZIG and S. EPSTEIN (*Monatsh.*, 1908, 29, 661—675).—In the constitution ascribed to reso flavin by Herzig and Tscherne (this vol., i, 547) there remains doubtful whether one of the hydroxyls occupies the position 2 or 4.



The analogy of the formation of reso flavin from 3:5-dihydroxybenzoic acid with that of flavellagic acid from gallic acid makes it probable that the hydroxyl in question occupies position 4, reso flavin having therefore the annexed constitution.

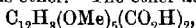
The behaviour of reso flavin when fused with potassium hydroxide is now found to be in agreement with this view of its constitution. Barth and Goldschmiedt (Abstr., 1879, 930) showed that ellagic acid, on fusion with potassium hydroxide, yields hexahydroxydiphenyl, pentahydroxydiphenylmethylolide (Graebe, Abstr., 1903, i, 262; Perkin and Nierenstein, Trans., 1905, 87, 1412) being formed intermediately. Reso flavin, on the other hand, when treated in the same manner, yields 3:5-dihydroxybenzoic acid, m. p. 237—240° (232—233°: Böttiger, this Journ., 1875, 567); the methyl ester, m. p. 163—165° (60°: Meyer, Abstr., 1901, i, 628). 3:5-Dimethoxybenzoic acid, m. p. 185—186° (175—176°: Tiemann and Streng, Abstr., 1882, 51); the methyl ester, m. p. 42—44° (81°: Meyer, Abstr., 1888, 148).

As ellagic acid, which contains two pyrogallol groups, yields hexahydroxydiphenyl, and, as the resorcinol nucleus is more stable than the pyrogallol nucleus towards fused alkalis, a substance having the annexed constitution must under the same conditions yield pentahydroxydiphenyl.



The degradation of reso flavin to 3:5-dihydroxybenzoic acid must be explained, therefore, by the presence of a resorcinol and a hydroxy-quinol grouping, the latter being more unstable towards the fused alkali than the carboxyl attached to the resorcinol portion of the complex.

When heated on the water-bath with alcoholic sulphuric acid, the ether ester of reso flavin yields a white substance, which, on treatment with diazomethane, forms trimethylreso flavin, and hence is the hydrolysis product of this ether. The ether acid,



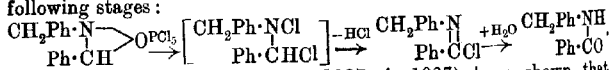
and trimethylreso flavin must be formed intermediately. In agreement with this, methyl 2:3:4:4':5':6'-hexamethoxydiphenyl-6:2'-carboxylate (Herzig and Pollak, this vol., i, 546), when treated with alcoholic sulphuric acid in the same manner, yields a yellow, crystalline substance, m. p. above 300°, which contains a slightly smaller percentage of methoxyl than tetramethylellagic acid, into which it is converted by the action of diazomethane.

G. Y.

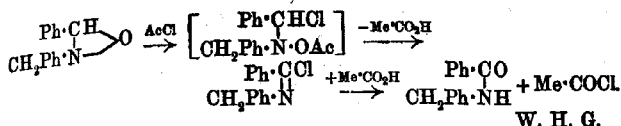
Nature and Constitution of Rhizocholic Acid. HUOT, SCHRÖTTER and RICHARD WEITZENBÖCK (*Monatsh.*, 1908, 29, 749—751).—It is now found that the acid obtained by the successive action of sulphuric and nitric acids on cholesterol, cholic acid, camphor, or turpentine oil, and previously termed rhizocholic acid (this vol., i, 532, 636), is benzenepentacarboxylic acid. When heated it sublimes, losing carbon dioxide and forming pyromellitic anhydride. It is considered that the pentacarboxylic acid is a secondary product, formed by oxidation of primarily liberated carbon. Its formation, therefore, cannot be looked upon as evidence of any relationship between cholesterol and cholic acid, on the one hand, and the terpenes, on the other. G. Y.

Action of Hydrogen Persulphide on Organic Compounds. HEINRICH BRUNNER and V. VUILLEUMIER (*Chem. Zentr.*, 1908, ii, 588; from *Schweiz. Woch. Chem. Pharm.*, 1908, 43, 436—438).—The authors have obtained the following substances by adding hydrogen persulphide to a solution of the organic compound in carbon disulphide, chloroform, or benzene. From *benzaldehyde*, long, colourless, prismatic crystals of the composition $(\text{Ph} \cdot \text{COH})_2, \text{H}_2\text{S}_3$, which decompose at 28° into sulphur, hydrogen sulphide, and benzaldehyde. *Anisaldehyde*, similar crystals of the composition $(\text{OMe} \cdot \text{C}_6\text{H}_4 \cdot \text{COH})_2, \text{H}_2\text{S}_3$, decomposing at 18° . *Cinnamaldehyde* gives a solid, non-crystallisable product, $\text{C}_9\text{H}_9\text{O}_3, \text{H}_2\text{S}_3$. *Benzoquinone*, a violet powder of the composition $(\text{C}_6\text{H}_4\text{O}_2)_2, \text{H}_2\text{S}_3$, which is possibly a mixture of $(\text{C}_6\text{H}_4\text{O}_2)_2, \text{H}_2\text{S}_3$ and $(\text{C}_6\text{H}_4\text{O}_2)_2, \text{H}_2\text{S}_2$. Piperidine in chloroform gives a red solution with sulphur, which, on addition of carbon disulphide, yields $\text{C}_5\text{H}_{11}\text{N} \cdot \text{CS} \cdot \text{S} \cdot \text{NH}_2, \text{H}_{11}$. Many aldehydes and ketones simply cause the precipitation of white, prismatic sulphur. The compounds obtained with strychnine and brucine were found to be identical with those described by Hoffmann and Schmidt (*Abstr.*, 1877, ii, 789, 905). J. V. E.

Molecular Rearrangement of *N*-Benzylaldoxime. MITSURU KUHARA (*J. Coll. Sci. Tokyo*, 1908, 25, xviii, 1—4).—The author has succeeded in isolating benzoylbenzylamine iminochloride (compare Pechmann and Heinze, *Abstr.*, 1897, i, 515) as an intermediate product of the rearrangement of *N*-benzylbenzaldoxime by the action of phosphorus pentachloride, and in converting the iminochloride into benzylbenzamide by the action of water. The transformation of *N*-benzylbenzaldoxime into benzylbenzamide under the influence of phosphorus pentachloride, therefore, probably takes place through the following stages:



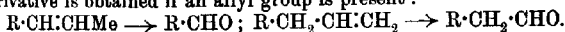
Kuhara and Kainosho (*Abstr.*, 1907, i, 1027) have shown that phenylbenzamide is formed by the action of acetic acid on chlorobenzylideneaniline. It is therefore probable that the rearrangement of *N*-benzylbenzaldoxime by the action of acetyl chloride takes place thus:



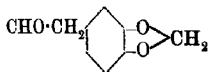
β-p-Methoxyphenylpropaldehyde obtained from the isomeric *α-p*-Methoxyphenylpropylene-*αβ*-glycols [*α-p*-Methoxyphenylpropane-*αβ*-diols]. LUIGI BALBIANO (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 259—269).—According to Tiffeneau and Daufresne (Abstr., 1907, i, 701), the *β*-modification of *α-p*-methoxyphenylpropylene-*αβ*-glycol (compare Balbiano, Abstr., 1907, i, 522) yields *p*-methoxyphenylacetone when dehydrated by means of 20% sulphuric acid. The author shows, however, that the compound is not a ketone, but *β-p*-methoxyphenylpropaldehyde, which is also obtained by dehydrating the isomeric *α*- and *β*-glycols by means of zinc chloride (compare Balbiano and Paolini, Abstr., 1906, i, 186). When either method of dehydrating the glycol is used, this aldehyde is accompanied by a small quantity of a dimeric form, $\text{OMe} \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \begin{smallmatrix} \text{O} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{O} \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{O} \end{smallmatrix} \text{CH} \cdot \text{C}_6\text{H}_4 \cdot \text{OMe}$ (?), which crystallises from alcohol in microscopic prisms, m. p. 181—182°, has the normal molecular weight in freezing benzene, and exhibits no aldehydic properties. When treated with benzenesulphonylhydroxamic acid, the *α*- or *β*-form of *β-p*-methoxyphenylpropaldehyde yields a small amount of the corresponding hydroxamic acid, which was separated as copper salt, $\text{C}_{10}\text{H}_7\text{O}_5\text{NCu}$; when dissolved in dilute hydrochloric acid, this salt gives an intense violet coloration with ferric chloride, whilst, on hydrolysis, it yields anisic acid and a small quantity of a substance, $\text{C}_6\text{H}_7\text{O}_4\text{N}$, crystallising in shining laminae, m. p. about 150°.

T. H. P.

Constituents of Ethereal Oils. Homopiperonal and its Derivatives. FRIEDRICH W. SEMMLER and KONRAD BARTLET (*Ber.*, 1908, 41, 2751—2753).—To distinguish between the aromatic constituents of ethereal oils containing an allyl or a propenyl group in the side-chain, the authors oxidise the substance with ozone, whereby rupture at the double linking occurs, with the formation of an aldehyde if a propenyl group is present, whilst the corresponding homoderivative is obtained if an allyl group is present:



Homopiperonal (annexed constitution), m. p. 69°, b. p. 143—144°/10 mm., D_{20}^{20} 1.295, n_D^{20} 1.57117, is obtained by passing ozone through a solution of safrole in benzene mixed with a small quantity of water, distilling the product



in steam to remove benzene and unchanged safrole, and extracting the non-volatile aldehyde with ether. The *semicarbazone*, m. p. 189°, *oxime*, m. p. 124—125°, b. p. 180—181°/10 mm., and *nitrile*, b. p. 153—156°/10 mm., D_{20}^{20} 1.231, n_D^{20} 1.53698, are described. Hydrolysis of the last by 10% alcoholic potassium hydroxide yields *homopiperonylic acid*, $\text{C}_9\text{H}_8\text{O}_4$, m. p. 127°, the *methyl ester* of which has b. p. 153—155°/10 mm.,

D_{20} 1.246, n_D 1.534, and is converted by sodium and absolute alcohol into homopiperonyl alcohol, $C_9H_{10}O_3$, b. p. $156^\circ/10$ mm., n_D 1.54780. By reduction, the nitrile yields the amine, $C_9H_{11}O_3N$, b. p. $146-148^\circ/10$ mm., D_{20} 1.225, n_D 1.5620, of which the picrate has m. p. 160° (decomp.). C. S.

Quinone Formation. RAYMOND VIDAL (*Chem. Zentr.*, 1908, ii, 240; from *Mon. Sci.*, 1908, [iv], 22, i, 368—372).—Quinone formation is considered as supporting the views expressed (Abstr., 1907, i, 1020) on the nature of the benzene ring. J. V. E.

Action of Piperidine on *l*-Pinene Chloroxime. P. G. GOLUBEFF (*J. Russ. Phys. Chem. Soc.*, 1908, 40, 1015—1018).—The action of piperidine on the chloroxime of *l*-pinene isolated from the ethereal oil of the Siberian fir proceeds in two directions: (1) $C_{10}H_{15}NOCl + C_5H_{11}N = HCl + C_{10}H_{15}NO \cdot C_5H_{11}N$, yielding a compound identical with that given by *d*-pinene from American turpentine;

(2) $C_{10}H_{15}NOCl + C_5H_{11}N = HCl + C_5H_{11}N + C_{10}H_{15}ON$, yielding the nitrosopinene obtained by the action of diethylamine on *d*-pinene chloroxime. T. H. P.

Essential Oil of French Parsley and the Contained Ether, 2:3:4:5-Tetramethoxy-1-allylbenzene. HERMANN THOMS (*Ber.*, 1908, 41, 2753—2761. Compare Abstr., 1904, i, 47).—Experiments are in progress to ascertain whether the climate, the conditions of cultivation, or the degree of ripeness of the seeds, accounts for the fact that the essential oil of French parsley contains a large quantity of myristicin and a small amount of apiole, whilst the oil from German parsley is rich in the latter.

A large quantity of the oil of French parsley has been freed from phenols and acids, and the resulting liquid separated into two fractions, b. p. $160-165^\circ/15$ mm. and $165-170^\circ/15$ mm. The presence of myristicin in both is proved by the formation of dibromomyristicin dibromide (Abstr., 1904, i, 47), but a Zeisel estimation indicates the presence of a substance richer in methoxyl groups. This substance, ultimately isolated by the aid of solid carbon dioxide, has been proved to be 2:3:4:5-tetramethoxy-1-allylbenzene, m. p. 25° , D_{25}^{20} 1.087, n_D^{25} 1.51462. The presence of the allyl group is indicated by the dispersion and the molecular refraction, and by the formation of a red coloration with phloroglucinol and hydrochloric acid (compare Kobert, *Zeitsch. anal. Chem.*, 1908, 47, 711). The positions of the methoxyl groups are determined by oxidation with potassium permanganate, whereby 2:3:4:5-tetramethoxybenzoic acid, m. p. 87° , is obtained, and also by successive treatment with alcoholic potassium hydroxide (which changes the allyl to a propenyl group), reduction with sodium and alcohol, and nitration of the product, resulting in the formation of 4-nitro-2:3:5-trimethoxy-1-propylbenzene (Abstr., 1903, i, 558). By nitration, the methoxyl group in position 4 has been replaced by a nitro-group (compare Thoms and Herzog, Abstr., 1903, i, 415).

The same operations on the above-mentioned fractions yield the same compounds, as also does the oil from French parsley seeds grown in Dählem. C. S.

Ethereal Oil from *Salvia sclarea*. ROURE-BERTRAND FILS (*Chem. Zentr.*, 1908, ii, 323; from *Wiss. ind. Ber. Roure-Bertrand Fils*, 1908, [ii], 7, 10—11).—The oil distilled in 1904 (*Chem. Zentr.*, 1906, ii, 535) has been saponified, and *l*-linalool is found to be the chief constituent, together with some resinous substances which render it insoluble in 96% alcohol, acetone, methyl alcohol, &c.

J. V. E.

Resin of *Antiaris toxicaria*. ADOLPH WINDAUS and A. WELSCH (*Arch. Pharm.*, 1908, 246, 504—508. Compare de Vrij and Ludwig, *Sitz. Wien. Akad.*, 1868, 57, 56, and Kiliani, *Abstr.*, 1897, i, 91).—The portion of the latex of *Antiaris toxicaria* soluble in light petroleum is composed principally of the cinnamic ester of α -amyrin, $C_{39}H_{56}O_2$, m. p. 176°, $[\alpha]_D^{25} + 78.45'$ (in chloroform), which crystallises from methyl alcohol in long, thick, colourless needles. From the products of hydrolysis of the amorphous portion of the resin, α -amyrin, cinnamic acid, and some stearic acid were obtained. No β -amyrin seems to be present in the resin.

T. A. H.

Decomposition of Amygdalin by Emulsin. KARL FEIST (*Arch. Pharm.*, 1908, 246, 509—510. Compare this vol., i, 437, and Rosenthaler, *ibid.*, i, 817).—Rosenthaler's observation, that emulsin is capable of forming *d*-benzaldehydecyanohydrin from benzaldehyde and hydrocyanic acid, is, contrary to that author's opinion, in reality a confirmation of Feist's statement that *d*-benzaldehydecyanohydrin is the first product of the action of emulsin on amygdalin, since the action probably reaches equilibrium in the two directions: *d*-benzaldehydecyanohydrin + emulsin \rightleftharpoons benzaldehyde + hydrocyanic acid + emulsin.

T. A. H.

New Glucoside (Erytaurin) obtained from the Common Century. HENRI HÉRISSEY and L. BOURDIER (*J. Pharm. Chim.*, 1908, [vi], 27, 252—255).—The glucoside, obtained from the plant by a lengthy extraction process, crystallises in small, colourless prisms on addition of ether to its solution in a mixture of chloroform and alcohol. It is bitter to the taste, has $[\alpha]_D - 131.6^\circ$ to -134.4° , does not reduce Fehling's solution, but gives a blue coloration with a mixture of ferric chloride and potassium ferricyanide. Erytaurin is slowly hydrolysed by emulsin, yielding dextrose and a pale yellow precipitate.

T. A. H.

Glucosides of *Linaria*. TIMOTHÉE KLOBB (*Bull. Soc. chim.*, 1908, [iv], 3, 858—872. Compare *Abstr.*, 1907, i, 864; ii, 123).—The yield of linarin from the dried flowers of *Linaria vulgaris* amounts to 1.5–2.8%; it may be recrystallised from 50% acetic acid. Saturated solutions in boiling alcohol contain 0.30% of linarin; in cold alcohol,

0.24%; in boiling 50% acetic acid, 0.57%, and in the cold acid, 0.36%. The glucoside apparently forms an additive compound with phenol, and is readily absorbed from solution by animal charcoal. β -Linarin has m. p. 257—260°, and is more soluble than linarin in 50% acetic acid.

α -Pectolinarin is straw-yellow and amorphous, but sometimes forms hard, almost white, semi-crystalline aggregates. Saturated solutions in alcohol at 80° contain 0.582% of pectolinarin; in cold alcohol, 0.097%; in water at 100°, 0.24%. Pectolinarin is rapidly hydrolysed by dilute acids at 85°, giving a mixture of the two phenols. Both linarin and pectolinarin, when hydrolysed by concentrated hydrochloric acid, give linaric phenol only.

Linaric phenol crystallises from glacial acetic acid in needles or orange-red prisms, the latter containing both water and acetic acid of crystallisation, which are lost at 120—130°. Its m. p. is 277—279°, not 245° as stated previously; it gives a greenish-black coloration with alcoholic ferric chloride, and a greenish-grey precipitate with chloroform and potassium hydroxide, becoming reddish-brown on exposure to the air. It forms an *acetate*, $C_{19}H_{11}O_7Ac$, m. p. 248—250°.

Anhydrolinaric phenol is quite stable towards alkali hydroxides (difference from linaric phenol), gives a violet-brown coloration with alcoholic ferric chloride, a bright red coloration with chloroform and potassium hydroxide, and a brown coloration in Liebermann's reaction. The *acetate* forms colourless needles, m. p. 198—200°, and the *benzoate*, $C_{19}H_{10}O_6(COPh)_2$, short, white needles, m. p. 199—201°. E. H.

Oleoeuropein; a New Glucoside obtained from the Olive, Olea Europa. ÉMILE BOURQUELOT and J. VINTILESIO (*Compt. rend.*, 1908, 147, 533—535; *J. Pharm. Chim.*, 1908, [vi], 28, 303—314). [Compare Power and Tutin, *Trans.*, 1908, 93, 891, 904.]—Bourquelot's biological test (*Abstr.*, 1902, ii, 55) for the detection of glucosides shows the presence of a glucoside, *oleoeuropein*, hydrolysable by emulsin in the fruits, leaves, and bark of the olive. This substance, which was only obtained as a slightly yellow powder, possesses a bitter taste, is readily soluble in cold water or warm alcohol, insoluble in ether, and has $[\alpha]_D - 5.166^\circ$. It reduces Fehling's solution on boiling, and its solution in water is coloured yellow by alkalis, red by sulphuric acid, and green by ferric chloride. The glucoside is hydrolysed by emulsin or by boiling with acids, yielding dextrose. Emulsin also occurs in the leaves and fruit of the olive. T. A. H.

***l*-Arabinose and Dextrose as Inversion Products of Saponin from the Leaves of *Polyscias nodosa*, Forst.** A. W. VAN DER HAAR (*Pharm. Weekblad*, 1908, 45, 1184—1191).—Anhydrous saponin from the leaves of *Polyscias nodosa* has the empirical constitution $C_{25}H_{42}O_{10}$, and contains an arabinose complex. On inversion, it takes up water, yielding *l*-arabinose, dextrose, and sapogenin. A. J. W.

The Hydrocarbon Nucleus of Frangula emodin, Aloe-emodin, and Rhein. OTTO A. OESTERLE and ED. TISZA (*Arch. Pharm.*, 1908, 246, 432—436).—A continuation of work on the constitution of the emodins obtained from the bark of *Rhamnus frangula* and from aloes respectively, showing that the former is a β -methylantracene derivative as already formulated (this vol., i, 350), and the latter probably derived from α -methylantracene.

Frangula emodin, on distillation with zinc dust, yields β -methylantracene (compare Liebermann, this Journ., 1876, i, 251; Perkin and Hummel, *Trans.*, 1894, 65, 924).

The hydrocarbon obtained when aloe-emodin is distilled with zinc dust forms greenish-yellow leaflets, has m. p. 208—209°, fluoresces green in acetic acid, gives a blood-red *picric acid* compound, m. p. 145°, and is polymerised in benzene solution on exposure to sunlight, yielding a hydrocarbon, m. p. 256°, crystallising in colourless needles. Since these characteristics are different from those of β -methylantracene, the authors suggest that the hydrocarbon thus obtained from aloe-emodin is probably α -methylantracene.

Rhein on distillation with zinc dust yields anthracene (compare Hesse, *Abstr.*, 1900, i, 41; Tschirch and Heuberger, *Abstr.*, 1903, i, 107; Oesterle, *Abstr.*, 1904, i, 80).

T. A. H.

Pimpinellin. JOHANNES HERZOG and V. HÂNCU (*Arch. Pharm.*, 1908, 246, 402—414. Compare Heut, *Abstr.*, 1898, i, 598).—The preparation of pimpinellin from the root of *Pimpinella saxifraga* has been simplified, the characters of the substance definitely determined, and its oxidation by hydrogen peroxide studied. The results of this work indicate that pimpinellin is a naphthalene derivative.

Pimpinellin, $C_{13}H_{10}O_5$, m. p. 119°, obtained to the extent of 0.5% by extracting the ground root with benzene and treating the concentrated extract with light petroleum, crystallises in glancing, white needles, sublimes with slight decomposition, and becomes yellow on long exposure to light. It is probably a lactone, contains two methoxyl groups, and, on oxidation with hydrogen peroxide, furnishes a tribasic acid, $C_9H_6O_8$, m. p. 220° (decomp.). This crystallises in short, thick needles from acetic acid, is readily soluble in alcohol, less so in water, and can be sublimed, forming, then, long needles recalling those of phthalic anhydride, which it resembles in giving a fluorescein reaction. The acid contains no methoxyl groups; it yields a well-crystallised pyridine salt, m. p. 179° (decomp.), and this reacts with diphenylcarbamyl chloride (compare this vol., i, 268) to form the diphenylated acid amide, $C_6H_5O_2(CO \cdot NPh_2)_3$, m. p. 224.5—225.5°, which separates from boiling alcohol or toluene in splendid pale yellow crystals.

The second crystalline product isolated by Heut (*loc. cit.*) from the roots could not be obtained, although evidence of the existence of a minute quantity of a second substance, possibly a decomposition product of pimpinellin, was observed.

T. A. H.

Formation and Chemistry of Anthocyanins. LEOPOLD VON PORTHEIM and EMIL SCHOLL (*Ber. deut. bot. Ges.*, 1908, 26a, 480—483).—Pure solutions of unstable, plant-colouring matters, such as that of

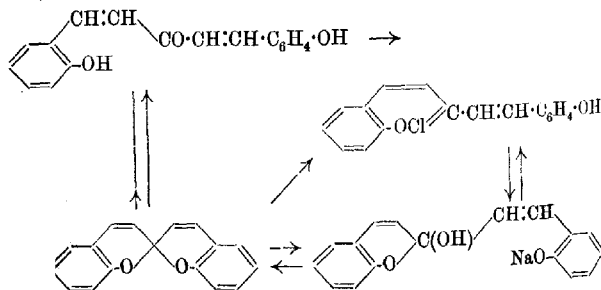
beetroot, can be prepared by dialysing the expressed juice or an aqueous extract of the plant (compare Weigert, Abstr., 1896, i, 337).

The colouring matter of the testa of the seed of *Phaseolus multiflorus* is insoluble in water. An alcoholic extract of the testa deposits on slight concentration a small quantity of a yellow substance, crystallising in microscopic bundles of needles. The mother liquor from this deposits no more crystalline matter on further concentration, but, when boiled with a few drops of hydrochloric acid, yields a reddish-violet liquid, which, after some days, deposits ruby-red, microscopic crystals of a substance, readily soluble in alcohol, and turning blue on addition of ammonia.

The mother liquor referred to above may also be separated into three fractions by treatment with ether. Of these, (A) is a viscid, brown mass, which yields the crystalline, red colouring matter on hydrolysis by acids; (B) a reddish-violet, amorphous product, and (C) a brownish-yellow residue, insoluble in water.

T. A. H.

Cyclic Oxonium Salts from Disalicylideneacetone, and spiroPyran Derivatives. HERMAN DECKER and HEINRICH FELSER (*Ber.*, 1908, 41, 2997—3007).—Tiemann and Kees' disalicylideneacetone (dicoumarone-ketone) (Abstr., 1885, 1073) is very easily obtained, in the form of the alkali salt, by adding alcoholic potassium hydroxide (3—4 mols.) to an alcoholic solution of salicylaldehyde (2 mols.) and acetone (rather less than 1 mol.). By treating an alcoholic solution of the ketone with hydrogen chloride or 50% sulphuric acid, a red solution of the 2-*o*-hydroxystyrylbenzopyrylium salt is obtained, from which ferric chloride precipitates the dark red ferrichloride, $C_{17}H_{13}O_2Cl_4Fe$, m. p. 180°. The very intense colour of these oxonium salts is due, in part, to the presence of the *o*-hydroxystyryl group. The red oxonium chloride gives with 10% sodium hydroxide a yellow solution of the sodium salt of 2-*o*-hydroxystyrylbenzopyranol-2, $C_6H_4 \begin{smallmatrix} \text{CH:CH} \\ \text{O} \end{smallmatrix} \text{C(OH):CH:CH} \cdot C_6H_4 \cdot ONa$, from which the carbinol itself cannot be isolated, since the carbinol and the phenolic hydroxyl groups eliminate water, and the stable, colourless, crystalline dibenzospiropyran, m. p. 102°, is formed, just as the carbinols of other cyclic oxonium salts react with alcohols to give ethers (Decker and



von Fellenberg, Abstr., 1907, i, 950). By treatment with concentrated acids, one pyran ring of the *spiropyran* derivative is readily ruptured, yielding the coloured 2-*o*-hydroxystyrylbenzopyrylium salt, whilst warm alcoholic sodium hydroxide opens both rings, with the regeneration of disalicylidineacetone.

The constitutions and the transformations of the preceding compounds are indicated in the preceding scheme.

These changes are not readily explicable by Bülow's benzopyranol-(4) formula (Abstr., 1901, i, 400, 559; 1902, i, 113).

Perkin, Robinson, and Turner (Trans., 1908, 93, 1088), criticising Decker's formula of the benzopyrylium salts, contend that with this formula these should not be so highly coloured as they actually are, since the linkings in the skeleton formula are in a similar position to those in quinoline; they suggest an alternative *o*-quinonoid formula. The authors argue that this formula does not explain the formation of benzopyrylium salts from coumarin, the ready hydrolysis of the carbinol, the colour of the salts, and the absence of colour in the carbinol. It is known that in acridinium and xanthylum salts the replacement of the NMe group by O in the ring intensifies the colour. Just the same difference occurs with benzopyrylium and quinolinium salts, and the deeper colour of the former is regarded as a proof that they are oxonium salts isologous with quinolinium salts.

2-*o*-Hydroxystyrylbenzopyrylium chloride, $C_{17}H_{13}O_3Cl \cdot H_2O$, forms red needles with a bronze-green reflex, and, from its moderately-concentrated solution, other crystalline salts and double salts can be obtained. Dibenzo*spiropyran* is formed when disalicylideneacetone is kept above its m. p., 168° , for ten minutes. C. S.

Brazanquinones. STANISLAUS VON KOSTANECKI and VICTOR LAMPE (*Ber.*, 1908, 41, 2800—2802).—As catechone trimethyl ether yields a well-crystallised nitro-derivative, the authors have investigated the action of nitric acid on the closely-allied 2:7:8-trimethoxybrazanquinone (this vol., i, 672), and find that it gives a trinitro-derivative; similarly, 2-methoxybrazanquinone yields a dinitro-derivative.

Trinitro-2:7:8-trimethoxybrazanquinone, $C_{19}H_{11}O_{15}N_3$, prepared by adding the quinone to nitric acid (D 1.5), crystallises in orange-yellow needles, m. p. 275° (decomp.). 1:3(4)-*Dinitro-2-methoxybrazanquinone* forms small, stout crystals, m. p. 253 — 254° (decomp.). In order further to characterise 2-methoxybrazanquinone, the authors have converted it by reduction and simultaneous acetylation into 5:10-*diacetoxy-2-methoxybrazan*, which crystallises in white, prismatic needles, m. p. 195 — 196° . Its solution in concentrated sulphuric acid is green, and shows an intense dark green fluorescence.

5:10-*Diacetoxy-2:7:8-trimethoxybrazan* (Kostanecki and Lloyd, Abstr., 1903, i, 646) gives the same colour reaction. When boiled with alcoholic potash and the product treated with dimethyl sulphate, 5:10-diacetoxy-2-methoxybrazan yields 2:5:10-*trimethoxybrazan*, crystallising in white leaflets, m. p. 165° . This shows the same colour reaction with sulphuric acid as the compound just described, and its alcoholic solution exhibits a weak blue fluorescence. J. C. C.

Morphine. XIX. Relationship between ψ -apocodeine and apomorphine. LUDWIG KNOBB and FELIX RAABE (*Ber.*, 1908, 41, 3050—3054).— ψ -apocodeine (Abstr., 1907, i, 790) stands in the same relationship to apomorphine as codeine to morphine; it is thus the 3-methyl ether of apomorphine, and is identical with Pschorr, Jäckel, and Fecht's methyl ether (Abstr., 1903, i, 193). The crystals obtained from alcoholic solutions contain 1EtOH, and melt and decompose at 105°, after sintering at 100°. In alcoholic solution it has $[\alpha]_D^{15} - 90^\circ$ ($c = 0.84$), and in chloroform solution, -103° ($c = 2.6$). The dimethyl ether of apomorphine has not been obtained crystalline, even after distillation under reduced pressure. It has b. p. 195—205° in the cathode ray vacuum, and $[\alpha]_D^{15} - 148^\circ$ ($c = 1.6395$) in alcoholic solution. The hydriodide, $C_{19}H_{22}O_2NI$, forms pale yellow prisms, m. p. about 220°.

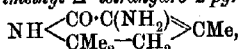
The rotatory power of methyl-apomorphine methiodide is $[\alpha]_D^{15} - 20^\circ$ ($c = 1.2573$) in methyl-alcoholic solution. Diacetylmethylapomorphine has m. p. 130°. J. J. S.

Protopine of Japanese Corydalis Roots (Corydalis Vernyi). K. MAKOSHI (*Arch. Pharm.*, 1908, 246, 401—402).—The root of this plant contains two alkaloids, the one, identical with protopine, occurring to the extent of 0.13%, and the other, obtained in the form of a yellow, crystalline mass and closely resembling dehydrocorydaline in appearance and properties, is present to the extent of 0.013%.

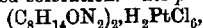
T. A. H.

Certain *o*-Amino- and *o*-Hydroxy-ketohydropyridinea. I. GALEAZZO PICCININI (*Atti R. Accad. Sci. Torino*, 1908, 43, 890—912).—The author describes various amino- and the corresponding hydroxy-tetrahydropyridine derivatives in which the amino- (or hydroxy-) group is in the 3 position, whilst the 2-position is occupied by what is most probably a carbonyl group.

3-Amino-4 : 6 : 6-trimethyl- Δ^3 -tetrahydro-2-pyridone,

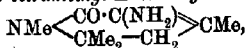


prepared by converting 4 : 6 : 6-trimethyl- Δ^3 -tetrahydro-2-pyridone-3-carboxylamide (compare this vol., i, 679) into the corresponding bromoamide by means of potassium hypobromite and then boiling with water, separates from benzene in friable, prismatic crystals, m. p. 130—131°, exhibiting a slight blue fluorescence, and yields an alkaline aqueous solution, which with ferric chloride gives an orange-red and, later, a blood-red coloration. Its *platinichloride*,



m. p. 235°, is decomposed when boiled with water, giving the corresponding hydroxy-compound (*vide infra*), $(\text{C}_8\text{H}_{14}\text{ON}_2)_2, \text{H}_2\text{PtCl}_6 + 2\text{H}_2\text{O} = 2\text{C}_8\text{H}_{14}\text{O}_2\text{N} + (\text{NH}_4)_2\text{PtCl}_6$. The hydroxy-compound is also formed by the action of nitrous acid on the amino-derivative.

3-Amino-1 : 4 : 6 : 6-tetramethyl- Δ^3 -tetrahydro-2-pyridone,



prepared from 1 : 4 : 6 : 6-tetramethyl- Δ^3 -tetrahydro-2-pyridone-3-

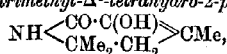
carboxylamide (*loc. cit.*), crystallises from ether in colourless or faintly yellow prisms, m. p. 60—61°, has an alkaline reaction, shows normal cryoscopic behaviour in benzene, and reacts with water or nitrous acid like the preceding compound. The *platinichloride*,
 $(C_9H_{16}ON_2)_2, H_2PtCl_6, H_2O$,
 has m. p. 196—197° (decomp.).

Both these amino-compounds are mono-acid bases rather weaker than ammonia; they do not absorb carbon dioxide from the air, but precipitate aluminium hydroxide from solutions of aluminium salts. The most striking properties of the bases are the ease with which they react with nitrous acid and the mobility of the amino-group towards hydrolysts, these being due to the presence of the group



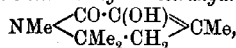
in a hydrogenated nucleus. They both readily reduce Fehling's solution, ammoniacal silver nitrate solution, &c.

3-Hydroxy-4 : 6 : 6-trimethyl- Δ^3 -tetrahydro-2-pyridone,



crystallises from water in shining leaflets, m. p. 143°, has a neutral reaction, gives a blue coloration with ferric chloride, and readily reduces Fehling's solution, ammoniacal silver nitrate solution, &c.

3-Hydroxy-1 : 4 : 6 : 6-tetramethyl- Δ^3 -tetrahydro-2-pyridone,

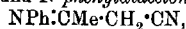


crystallises from alcohol in colourless needles, m. p. 95°, has the normal molecular weight in freezing benzene, gives a violet coloration with a little ferric chloride and an intense azure coloration with excess of the reagent, and readily reduces Fehling's solution, &c. T. H. P.

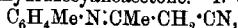
Bimolecular Nitriles. ERNST VON MEYER (*Chem. Zentr.*, 1908, ii, 591—594; from *Ber. K. Sächs. Ges. Wiss., Math.-phys. Kl.*, 1908, 60, 146—174. Compare *Abstr.*, 1895, i, 582—585).—The author has further investigated the behaviour of the so-called dinitriles which have long been shown (*loc. cit.*) to react as two tautomeric forms; diacetonitrile, for instance, either as iminoethylacetonitrile or β -aminocrotononitrile,

$$\begin{array}{c} Me \cdot C \cdot NH \\ | \\ CH_2 \cdot CN \end{array} \quad \text{or} \quad \begin{array}{c} Me \cdot C \cdot NH_2 \\ | \\ CH \cdot CN \end{array}$$

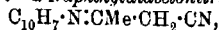
[With W. SCHUMACHER.]—*Action of Arylamines on Diacetonitrile.*—Aniline gives the compound *N*-phenyldiacetonitrile,



which crystallises in white needles, m. p. 115°; when treated with diazobenzene nitrate in pyridine solution, a *phenylhydrazone*, $NPh \cdot CMe \cdot C(N \cdot NHPh) \cdot CN$, is formed, crystallising in greenish-yellow leaflets, m. p. 149°, and this, when treated with hydrogen chloride, gives phenylhydrazoacyanoacetone. *N*-o-Tolyldiacetonitrile,



is prepared in a similar manner, and crystallises in leaflets, m. p. 73°. *N*-m-Tolyldiacetonitrile, white, prismatic needles, m. p. 109°. *N*-p-Tolyldiacetonitrile, white needles, m. p. 102°. *N*-Benzoyldiacetonitrile, white needles, m. p. 79°. α -Naphthyldiacetonitrile,



prepared from diacetonitrile and α -naphthylamine, crystallises in leaflets, m. p. 112°. *β -Naphthyl diacetonitrile*, brown leaflets, m. p. 172°. The three isomeric mononitroanilines and *o*-chloroaniline do not react with diacetonitrile, but *N*-m-chlorophenyl diacetonitrile,

$$\text{C}_6\text{H}_4\text{Cl}\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN},$$

was readily obtained as white needles, m. p. 136°, and *N*-p-chlorophenyl diacetonitrile as white leaflets, m. p. 114°. With the three phenylenediamines, the following compounds have been obtained: *o*-phenylenebisdiacetonitrile, $\text{C}_6\text{H}_4(\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN})_2$, prismatic leaflets, m. p. 136°; *m*-phenylenebisdiacetonitrile, prisms, m. p. 185°; *p*-phenylenebisdiacetonitrile, square leaflets, m. p. 222°. *p*-Hydroxyphenyl diacetonitrile, $\text{OH}\cdot\text{C}_6\text{H}_4\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN}$, crystallises in brown needles, m. p. 120°; *p*-ethoxyphenyl diacetonitrile, white needles, m. p. 138°. No combination takes place with *o*-aminobenzoic acid, but the other isomerides react, giving *m*-carboxyphenyl diacetonitrile,

$$\text{CO}_2\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN},$$

white, prismatic needles, m. p. 206°; *p*-carboxyphenyl diacetonitrile, needle-shaped crystals, m. p. 158°. All the above-mentioned compounds are decomposed by hydrogen chloride into acetic acid, ammonia, and arylamine.

[With H. LEHMANN].—*Action of Hydrazine Derivatives on Diacetonitrile*.—Carbamidodiacetonitrile, $\text{NH}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN}$, is produced from diacetonitrile and semicarbazide hydrochloride; it forms small, white plates, m. p. 165°, which re-melt at 220°; an isomeric modification crystallising in prisms, m. p. 134°, has been observed. Thiocarbamidodiacetonitrile, $\text{NH}_2\cdot\text{CS}\cdot\text{NH}\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN}$, long needles, m. p. 106°. With thiosemicarbazide hydrochloride an isomeric form is obtained which crystallises in rhombic plates, m. p. 132°, re-melting at 162°. Benzoylaminodiacetonitrile, $\text{COPh}\cdot\text{NH}\cdot\text{N}:\text{CMe}\cdot\text{CH}_2\cdot\text{CN}$, leaflets, m. p. 90°.

[With M. KLEINSTÜCK].—*Action of Aldehydes on Dinitriles* (compare Mohr, Abstr., 1898, i, 26).—2:6-Dimethyl-3:5-dihydropyridine, prepared from diacetonitrile and formaldehyde, crystallises in yellow tablets, m. p. 222°, which are oxidised by nitric acid to 3:5-dicyano-2:6-dimethylpyridine, slender, white needles, m. p. 112°. Diacetonitrile with acetaldehyde (or paraldehyde) in presence of hydrogen chloride yields 3:5-dicyano-2:4:6-trimethyldihydropyridine, needle-shaped crystals, m. p. 170°, which also becomes oxidised by nitric acid to 3:5-dicyano-2:4:6-trimethylpyridine, m. p. 111°. Benzodiacetonitrile and benzaldehyde (or hydrobenzamide) react in presence of hydrogen chloride, forming 3:5-dicyano-2:4:6-triphenyldihydropyridine, small, yellow plates, m. p. 268°; when treated with nitric acid, 3:5-dicyano-2:4:6-triphenylpyridine is formed, which crystallises in needles, m. p. 238°. In a similar manner, 3:5-dicyano-2:6-diphenyl-4-*o*-nitrophenyldihydropyridine is produced from *o*-nitrobenzaldehyde; it forms small, yellow plates, m. p. 258°. From benzodiacetonitrile and hexamethylenamine, 3:5-dicyano-2:6-diphenyldihydropyridine is obtained as small, yellow needles, m. p. 228°. With acetaldehyde or paraldehyde, 3:5-dicyano-2:6-diphenyl-4-methyldihydropyridine is obtained, needles, m. p. 267°.

[With W. HENNING].—*Condensation of Diacetonitrile* (compare

Holtzward, Abstr., 1889, 577).—The substance $C_8H_5N_3$, obtained from diacetonitrile by withdrawal of ammonia, is found to be 6-amino-3-cyano-2:4-dimethylpyridine, white needles, m. p. 222°, which, when treated with nitric acid, give 3-cyano-6-hydroxy-2:4-dimethylpyridine, needles, which melt above 260° (decomp.). The last-named substance gives, with phosphorus pentachloride, 6-chloro-3-cyano-2:4-dimethylpyridine, crystallising in thin leaflets, m. p. 65°; when boiled with methyl alcohol and sodium methoxide, 3-cyano-6-methoxy-2:4-dimethylpyridine is formed, needles, m. p. 96°, b. p. about 239°. This substance, when heated at 300°, changes to 3-cyano-1:2:4-trimethyl-6-pyridone, needle-shaped crystals, m. p. 125°. The above-mentioned chloro-compound (m. p. 65°), when reduced by zinc and hydrogen chloride, yields 3-cyano-2:4-dimethylpyridine, which forms colourless, prismatic crystals, m. p. 53°, b. p. 108°/15 mm., b. p. 218°; $C_8H_5N_2.HCl$, m. p. 187°; $C_8H_5N_2.HAuCl_4$, m. p. 172°; $C_8H_5N_2.HCl.HgCl_2$, m. p. 178°; $C_8H_5N_2.C_6H_5(NO_2)_2OH$, yellow prisms, m. p. 161°. When heated at 180° with alcoholic potassium hydroxide, lutidinamide is produced, which crystallises in needles with $\frac{1}{2}$ mol. H_2O , m. p. 191°; when heated with hydrogen chloride at 220°, 2:4-dimethylpyridine (ay-lutidine) is formed; hydrochloride, $C_8H_5Me_2N.HCl$; platinichloride, orange-red crystals, m. p. 208°. Contrary to Moir (Trans., 1902, 81, 10), the compound $C_8H_5ON_2$, which is isomeric with 3-cyano-6-hydroxy-2:4-dimethylpyridine (Holtzward, *loc. cit.*), is shown to be probably cyanodimethyl-6-pyridone; it gives with phosphorus pentachloride a compound, $C_8H_6N_2$, which is probably 2:4-dicyano-1:3-dimethylcyclobutane.

[With C. IRMSCHER].—Condensation of Dinitriles with β -Keto-carboxylic Esters and Unsaturated Ketones.—Condensation is incomplete with ketonic esters. From diacetonitrile and ethyl acetate in the presence of pyridine, there is obtained 3-cyano-2:6-dimethyl-4-pyridone, which crystallises in small, white needles, m. p. 280°. With benzodiacetonitrile in the presence of hydrogen chloride, the same ester gives 3-cyano-2-phenyl-6-methyl-4-pyridone, white leaflets, m. p. 244°. From unsaturated ketones and dinitriles, instead of dihydro-pyridine derivatives being produced, pyridine derivatives are obtained, owing to loss of two hydrogen atoms. Benzylideneacetophenone and diacetonitrile yield 3-cyano-4:6-diphenyl-2-methylpyridine, which crystallises in needles, m. p. 116°; $(C_{19}H_{14}N_2)_2.H_2PtCl_6$, golden-yellow leaflets. When heated with concentrated hydrochloric acid at 260°, it gives 4:6-diphenyl-2-methylpyridine-3-carboxylic acid, leaflets, m. p. 264°; the corresponding amide crystallises with 1 mol. H_2O , but the anhydrous substance, m. p. 216°. When the acid is distilled with soda-lime, 4:6-diphenyl-2-methylpyridine is produced, yellow needles, m. p. 156°. Potassium permanganate oxidises the acid compound to 4:6-diphenylpyridine-2:3-dicarboxylic acid, needles, m. p. 185° (decomp.). Diacetonitrile and anisylideneacetophenone with sodium ethoxide yield 3-cyano-4-p-methoxyphenylphenyl-6:2-methylpyridine, white needles, m. p. 157°. With piperonalacetophenone, 3-cyano-6-phenyl-4-methylenedioxyphenyl-2-methylpyridine, long, white needles, m. p. 238°; phenyl-p-tolylpropenone gives 3-cyano-4-phenyl-6-p-tolyl-2-methylpyridine, needles, m. p. 165°. Benzodiacetonitrile gives

3-cyano-2:4:6-triphenylpyridine, crystallising in needles, m. p. 220°; *p*-toluidiacetonitrile gives 3-cyano-4:6-diphenyl-2-*p*-tolylpyridine, needles, m. p. 185°. From anisylideneacetophenone and benzodiacetonitrile there is obtained: 3-cyano-2:6-diphenyl-4-*p*-methoxyphenylpyridine, needles, m. p. 181°; and from anisylideneacetophenone and *p*-toluidiacetonitrile, 3-cyano-6-phenyl-4-*p*-methoxyphenyl-2-*p*-tolylpyridine, needle-shaped crystals, m. p. 195°. Piperonalacetophenone yields 3-cyano-2:6-diphenyl-4-methylenedioxyphenylpyridine, m. p. 233°; 3-cyano-6-phenyl-2-*p*-tolyl-4-methylenedioxyphenylpyridine crystallises in needles, m. p. 245°. From cinnamylideneacetophenone, 3-cyano-2:6-diphenyl-4-cinnamylpyridine, needles, m. p. 184°; 3-cyano-6-phenyl-2-*p*-tolyl-4-cinnamylpyridine, needles, m. p. 181°. Benzylideneacetone, benzylidenecamphor, and benzylidenepyrrolone react in a different manner to benzylideneacetophenone.

[With W. SCHUMACHER.]—*Triazole Derivatives from Dinitriles.*—Diacetonitrile condenses with phenylazoinide, forming 1-phenyl-5-methyltriazole-4-carboxylic acid, which crystallises with $\frac{1}{2}$ H₂O, m. p. 111°; the anhydrous substance has m. p. 146°. Benzodiacetonitrile gives 4-cyano-1:5-diphenyltriazole, crystallising in nearly white needles, m. p. 242°, together with 1:5-diphenyltriazole-4-carboxylamide, yellow leaflets, m. p. 173°.

J. V. E.

Action of Methyl Iodide on 2:6-Substituted Pyridine-carboxylic Acids. RICHARD TURNAU (*Monatsh.*, 1908, 29, 845–852).

—An extension of the author's previous work on abnormal salts of betaines and pyridinecarboxylic acids (*Abstr.*, 1905, i, 546).

2-Methylpyridine-6-carboxylic acid yields a basic hydriodide, HI, C₇NH₅Me·CO·O·C₆NH₂Me·CO₂H, which crystallises from alcohol in long needles and from water in stout prisms, turning brown above 200°, and decomposing at 230°; the same salt is also produced by the interaction of methyl iodide and the anhydrous acid. When shaken with silver chloride, the hydriodide yields 2-methylpyridine-6-carboxylic acid and the normal hydrochloride, m. p. 201° (decomp.) (Pinner and Lewin, *Abstr.*, 1900, i, 409, give 138°).

2-Methylpyridine-6-carboxylic chloride, prepared by the action of thionyl chloride on the acid, forms slender, white needles, m. p. 195° (decomp.).

Quinoline-2-carboxylic acid does not react with methyl iodide at 100°, either with or without the presence of methyl alcohol. Pyridine-2:6-dicarboxylic acid does not react with methyl iodide at 100°, but in methyl-alcoholic solution gives the dimethyl ester. The author was unable to obtain a methiodide of 2-phenylcinchonic acid as described by Claus and Büttner (*Abstr.*, 1893, i, 731).

Nicotinic acid hydriodide is almost colourless and rather unstable; isonicotinic acid hydriodide forms slender, yellow needles, turning brown at 180°, and decomposing above 200°; both these are normal salts.

J. C. C.

Electrolytic Reduction of the Indoles. ORESTE CARRASCO (*Gazzetta*, 1908, 38, ii, 301–308).—Tetrahydrocarbazole, 2:3-dimethylindole, 2-methylindole, and 1-methylindole can be completely

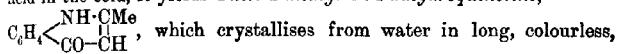
reduced electrolytically in an acid solution by means of lead electrodes separated by a porous diaphragm, but indole itself, although largely reduced to the corresponding indoline, undergoes secondary condensations or polymerisations apparently analogous to those occurring during the reduction of quinoline (compare Ahrens, Abstr., 1897, i, 368).

Thus tetrahydrocarbazole yields the carbazoline obtained by Graebe and Glaser (this Journ., 1872, 302); 2:3-dimethylindole gives 2:3-dimethylindoline, the *oxalate* of which, $C_{10}H_8N, H_2C_2O_4$, m. p. 134°, was prepared; 2-methylindole yields 2-methylindoline; 1-methylindole, 1-methylindoline, and indole, indoline. In the last case, a solid basic substance is also obtained, which is probably a polymeride of the hypothetical indolenine, $C_8H_4 \begin{smallmatrix} \text{CH}_2 \\ \diagup \quad \diagdown \\ \text{N} \end{smallmatrix} \text{CH}$.

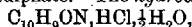
The electrolytic reducing action is related to the number and position of the alkyl groups in the pyrrole nucleus. Thus 2:3-methylindole is reduced more easily than 2-methylindole, and the latter more easily than 1-methylindole, further proof being so afforded of the great stability of the 1-indole derivatives compared with the 2- or 3-derivatives.

T. H. P.

Reduction of Nitro-compounds with Zinc Dust and Acetic Acid. II. GUSTAV HELLER and APOSTOLOS SOURLIS (*Ber.*, 1908, 41, 2692—2703. Compare this vol., i, 867).—When *o*-nitrophenyl-hydroxyethyl methyl ketone is reduced with zinc dust and 33% acetic acid in the cold, it yields 4-*keto*-2-methyl-1:4-dihydroquinoline,



hydrated needles, m. p. 77—78°. After remaining over sulphuric acid, it loses all but $\frac{1}{2}H_2O$. 2-Methylquinoline is formed on heating it with methyl sulphate. The *hydrochloride*,



forms small, white rods, which darken at 190°, and decompose at 202°. The *hydriodide*, $C_{10}H_9ON, HI, \frac{1}{2}H_2O$, separates in long, cherry-red rods, m. p. 167°. The *zincchloride*, $(C_{10}H_9ON)_2 \cdot HCl, ZnCl_2, H_2O$, has m. p. 211°. The *platinichloride*, $(C_{10}H_9ON)_4 \cdot H_2PtCl_6, 2H_2O$, forms lemon-yellow prisms, m. p. 207° (decomp.). The *dichromate* occurs in small, orange-yellow rods, m. p. 133—134° (decomp.). The *picrate* crystallises in lemon-yellow, six-sided rhombohedra, m. p. 173°. When the base is treated with phosphoryl chloride and phosphorus pentachloride, 4-chloro-2-methylquinoline (Conrad and Limpach, Abstr., 1887, 680) is formed. This crystallises with $1H_2O$, which is lost in a desiccator. 4-*Keto*-1:4-dihydroquinoline-2-carboxylic acid, prepared by oxidising the base with potassium permanganate in alkaline solution, crystallises in colourless needles, m. p. 167° (decomp.); on reduction with hydriodic acid and red phosphorus, it yields quinoline-2-carboxylic acid. When 4-*keto*-2-methyl-1:4-dihydroquinoline is reduced with zinc dust and hydrochloric acid, it yields 2-methylquinoline, of which the following salts are described and some inaccurate statements concerning them in the literature corrected. The hydrochloride (mentioned by the author as not having previously been described, but referred to by

Doebner and von Miller, Abstr., 1883, 602), colourless needles, m. p. 224° (decomp.); the dichromate, yellow needles, m. p. 110°; the methiodide darkens at 230°, m. p. 234° (decomp.) (Doebner and von Miller, Abstr., 1884, 184, give 195°); the zincchloride, colourless rhombohedra, darkens at 205° and decomposes at 245°; this salt is formed in the reduction of *o*-nitrophenylhydroxyethyl methyl ketone, and if this process is carried on with addition of 1 part of hydrochloric acid, a yield of 52% of the theoretical amount of 2-methylquinoline is obtained. The mercurichloride of 2-methylquinoline has m. p. 184° (Pictet and Bunzl, Abstr., 1889, 971, give 165.5°), and the picrate, m. p. 194°.

2-Methyldihydroquinoline, prepared by reducing 2-methylquinoline with hydrochloric acid and zinc dust, forms feathery crystals, m. p. 178°, and is bimolecular. It differs from the 2-methyldihydroquinoline described by Doebner (Abstr., 1898, i, 385), and the termolecular compound prepared by Ahrens (Abstr., 1897, i, 370). The *picrate* forms small, dark brown rods, which sinter at 95° and decompose at 110°.

J. C. C.

Constitution of Cyanine Dyes. EDUARD VONGERICHTEN and C. HÖFCHEN (*Ber.*, 1908, 41, 3054—3062. Compare Miethe and Book, Abstr., 1904, i, 622, 776).—The authors are in favour of Miethe and Book's constitutional formula for diethylisocyanine iodide (ethyl-red). Their support is based on the following considerations: (1) *Diethylchloroisocyanine iodide*, obtained from the ethioides of 6-chloroquinoline and 2-methylquinoline, has the formula $C_{24}H_{28}N_2ClI$. It crystallises from alcohol in a mixture of dark reddish-violet, flat needles, and of green crystals resembling ethyl-red, or from chloroform in homogeneous, green crystals. (2) Ethyl-red is a tertiary amine, since it forms a *methiodide*, $C_{24}H_{28}N_2I_2$, which crystallises in minute, brown needles, m. p. about 230°. It is decomposed by solution in alcohol, yielding ethyl-red. (3) In the condensation of the ethioides of quinoline and 2-methylquinoline, the methyl group of the latter is concerned, as when two hydrogen atoms of this methyl group are replaced by the benzylidene group or by two methyl groups, the condensation with quinoline ethioidide does not take place.

The action of the alkali on the 2-methylquinoline ethioidide is supposed to give the ammonium hydroxide derivative, which is then transformed into the carbinol base, and this by loss of water gives the *isobase*, $C_8H_4 \begin{smallmatrix} \text{NEt} \cdot \text{C} \cdot \text{CH}_2 \\ \text{CH} : \text{CH} \end{smallmatrix}$, which then condenses with the quinoline ethioidide. The presence of this grouping in ethyl-red is demonstrated by the formation of 1-ethyl-2-quinolone by the oxidation of ethyl-red, or its chloro-derivative with excess of cold potassium ferricyanide solution.

In the preparation of ethyl-red, the yield of dye is considerably lessened when more than two molecular proportions of potassium hydroxide are used for one of 2-methylquinoline methiodide and two of quinoline ethioidide.

6-Chloroquinoline *nitrate* forms colourless, glossy needles, m. p. 176°; the *chromate*, which can be used for purifying the base, golden-yellow

needles, m. p. 168°, and the *ethiodide*, C_9H_8ClN, EtI , yellow plates, m. p. 168—169°.

*α-iso*Propylcinchoninic acid has m. p. 150°, not 146°, and *α-iso*propylquinoline methiodide, $C_{13}H_{16}NI$, m. p. 182°.

Ethyl-red is not formed when 2-methylquinoline ethiodide is added to a hot solution of quinoline ethiodide and alcoholic potash solution, but is formed when cold solutions are used. This indicates that 1-ethyl-2-quinolone and 1-ethyltetrahydroquinolone, which are formed by the action of hot alkali on the carbinol base, cannot be intermediate products in the formation of the dye.

Cold aqueous potassium hydroxide reacts with 2-methylquinoline ethiodide, yielding a base which is soluble in ether or benzene, and the base appears to be a carbinol base, as it yields 2-methylquinoline ethochloride and water when its dry benzene solution is treated with hydrogen chloride.

1-Methyl-4-quinaldone reacts with phosphorus pentachloride, yielding a crude product, which condenses with 2-methylquinoline ethiodide and alkali to ethyl-red.

J. J. S.

Formation of 4-Oxycarbostryl from *o*-Nitrobenzoylactic Acid. KOICHI MATSUBARA (*J. Coll. Sci. Tōkyō*, 1908, 25, xvii, 1—3).—4-Oxycarbostryl (or 2:4-dihydroxyquinoline) is formed by adding an ammoniacal solution of *o*-nitrobenzoylactic acid to an aqueous solution of ferrous sulphate to which an excess of ammonia has been added.

W. H. G.

Action of Acid Esters on Quinoline Bases. JOHANN SPADY (*Ber.*, 1908, 41, 2902—2907).—On heating methyl salicylate with 2-methylquinoline at 180°, a quinaldine dye, $C_{23}H_{20}N_2$, phenol, and carbon dioxide are formed. The reaction is analogous to that between alkyl salicylates and primary or secondary amines studied by Tingle. The dye forms microscopic, yellow prisms from benzene, m. p. 190—191°; it dyes silk or cotton yarn similarly to quinaldine-yellow; it yields a sparingly soluble, crystalline hydrochloride and sulphate, and dissolves in concentrated sulphuric acid to a yellow solution having a bluish-green fluorescence.

Methyl salicylate reacts with quinoline at 150°, and energetically at 180—190°, forming a compound, $C_{20}H_{22}O_2N_{2,2}H_2O$, crystallising in minute, glistening, orange needles, which sinter at 281—282°, and melt and decompose at a higher temperature. It is soluble in water and alkaline hydroxides, and its solutions, particularly in alcohol or benzene, have a green fluorescence.

E. F. A.

Quinonoid Compounds. XVII. *meri*-Quinoneimines. II. RICHARD WILLSTÄTTER and JEAN PICCARD (*Ber.*, 1908, 41, 3245—3252. Compare this vol., i, 475).—as-*meri*-Quinonedimethyldiimonium nitrate, $NO_2 \cdot NH_2 \cdot \langle \bigcirc \rangle \cdot NMe_2 \cdot NO_2 \cdot NH_2 \cdot \langle \bigcirc \rangle \cdot NMe_2$, is prepared by the action of nitrous gases on a solution of *p*-phenylene-dimethyldiamine in alcohol and nitric acid, cooled by a mixture of ice

and calcium chloride. The leaflets are green by transmitted light, and show a copper metallic lustre; the solution is red.

Potassium ferricyanide in aqueous solution, when stirred into an acetic acid solution of the phenylenedimethyldiamine, forms green, rectangular plates of *as-meri-quinonedimethyldi-imonium ferricyanide*, $\text{H}_2\text{Fe}(\text{CN})_6 \cdot \text{NMe}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{NH}_2 \cdot 2\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{NMe}_2$.

Wurster and Schobig obtained a blue dye from tetramethyl-phenylenediamine which could only be analysed as the ferricyanide (Abstr., 1880, 111). Two *meri-quinonoid* compounds have been obtained by the authors, the blue salt, $\text{C}_{10}\text{H}_{16}\text{N}_2 \cdot \text{H}_3\text{Fe}(\text{CN})_6$, and the new violet salt, $3\text{C}_{10}\text{H}_{16}\text{N}_2 + \text{H}_2\text{Fe}(\text{CN})_6$ minus H_2 .

From benzidine, two *meri-quinonoid* chromates have been obtained: the first, $\text{NH} \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdot 4\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NH}_2 \cdot 4\text{H}_2\text{CrO}_4$, is formed when a chromic acid solution is treated at 0° with an excess of an acetic acid solution of benzidine. It is blue, the other is brownish-violet, and is obtained when excess of chromic acid is employed. It has the composition:

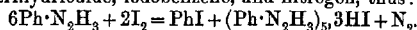
$\text{NH} \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdot 3\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NH}_2 \cdot 5\text{H}_2\text{CrO}_4$
(compare Willstätter and Kalb, Abstr., 1906, i, 996).

The quinonoid derivatives of tetramethylbenzidine prepared by Willstätter and Kalb (Abstr., 1904, i, 1050) have been re-examined by the new titration method with stannous chloride. The formula of the yellow *holo-quinonoid* disulphate has been confirmed; the green *meri-quinonoid* dichloride has the constitution:

$\text{NMe}_2\text{Cl} \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NMe}_2\text{Cl} \cdot \text{NMe}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NMe}_2 \cdot 6\text{H}_2\text{O}$.

W. R.

Action of Halogens and Hydrogen Halides on Phenylhydrazine. GEORG LOCKEMANN and E. WEINIGER (*Ber.*, 1908, 41, 3102—3108. Compare Fischer, this Journ., 1877, ii, 887; von Meyer, Abstr., 1887, 1042; Stollé, Abstr., 1903, ii, 100).—Iodine acts on phenylhydrazine dissolved in absolute ether, yielding pentaphenylhydrazine trihydriodide, iodobenzene, and nitrogen, thus:

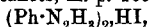


Bromine acts in a similar manner, but chlorine yields only the normal hydrochloride.

Pentaphenylhydrazine trihydriodide, $(\text{Ph} \cdot \text{N}_2\text{H}_3)_5 \cdot 3\text{HI}$, crystallises in thin, colourless leaflets, m. p. 137° . An alcoholic solution of the substance when treated with ether yields the *monohydriodide*,



crystallising in colourless leaflets, m. p. 165° . The *basic salt*,



is prepared by passing small quantities of hydrogen iodide into an ethereal solution of phenylhydrazine; it crystallises in small, colourless needles, m. p. 132° .

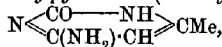
Pentaphenylhydrazine trihydrobromide, $(\text{Ph} \cdot \text{N}_2\text{H}_3)_5 \cdot 3\text{HBr}$, is obtained as a white, crystalline precipitate, m. p. 194 — 197° . Phenylhydrazine hydrobromide and the basic salt, $(\text{Ph} \cdot \text{N}_2\text{H}_3)_{1/2} \cdot \text{HBr}$, melt at 218° and 183° respectively. These differ from the m. p.'s recorded by Le Canu (Abstr., 1903, i, 778), Broche (Abstr., 1894, i, 562), and Ponzio (Abstr., 1906, i, 482).

W. H. G.

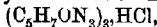
Action of Nitrous Esters on Hydrazine, Phenylhydrazine, and Benzylhydrazine in Alkaline Solution. ROBERT STOLLÉ (*Ber.*, 1908, 41, 2811—2813).—The author had already patented the same process as that since described by Thiele (this vol., ii, 940) for the preparation of azoimide. The best yields (80%) are obtained by boiling an alcoholic solution of hydrazine hydrate with amyl nitrite and sodium ethoxide. By neutralising hydrazine sulphate with aqueous potassium hydroxide and shaking the solution with alcohol, potassium hydroxide, and amyl nitrite, a 70% yield is obtained, and yields of 60% (of the hydrazine sulphate used) result when no alcohol is used. The author has also prepared isodiazobenzene salts by the action of ethyl nitrite on phenylhydrazine in presence of sodium or potassium ethoxide (compare Thiele, this vol., i, 927). The *silver* salt is white. Benzylhydrazine when treated with ethyl nitrite and sodium ethoxide in alcoholic solution yields, after some days, a mixture of sodium azoimide and sodium dibenzylhydrazine. J. C. C.

Pyrimidines. XXXVII. Synthesis of 4-Methylcytosine. CARL O. JOHNS (*Amer. Chem. J.*, 1908, 40, 348—355).—Of the two methyl derivatives of cytosine having the methyl group attached to carbon, the 5-methyl compound has already been prepared (Wheeler and Johnson, *Abstr.*, 1904, i, 624), and the 4-methyl compound is described in the present paper. When 6-oxy-2-ethylthiol-4-methylpyrimidine (obtained in an 80—90% yield by condensing ψ -ethylthiocarbamide hydrobromide with ethyl acetoacetate in aqueous sodium hydroxide) is heated with phosphoryl chloride, it is converted into 6-chloro-2-ethylthiol-4-methylpyrimidine, $\text{N} \begin{smallmatrix} \text{C(SET):N} \\ \text{CCl-CH} \end{smallmatrix} \text{CMe}$, b. p. 142°/15 mm., which, with sodium ethoxide, furnishes 6-ethoxy-2-ethylthiol-4-methylpyrimidine, $\text{N} \begin{smallmatrix} \text{C(SET):N} \\ \text{C(OEt)-CH} \end{smallmatrix} \text{CMe}$, b. p. 154°/20 mm.

When 6-chloro-2-ethylthiol-4-methylpyrimidine is heated with alcoholic ammonia under pressure, it yields 6-amino-2-ethylthiol-4-methylpyrimidine, $\text{N} \begin{smallmatrix} \text{C(SET):N} \\ \text{C(NH}_2\text{)-CH} \end{smallmatrix} \text{CMe}$, which forms crystals with a tetrahedral outline and a tendency to twinning, m. p. 115—116°. On evaporating this substance with concentrated hydrochloric acid, it gives 2-oxy-6-amino-4-methylpyrimidine (4-methylcytosine),



crystallising in anhydrous, slender, brittle prisms, which do not melt at 310°, but decompose slowly at higher temperatures. At 24°, 0.36 gram is dissolved by 100 c.c. of water. The *monohydrochloride* forms blunt, flat prisms with a hexagonal appearance; it sinters at 280°, and decomposes slowly above 300°. The basic *hydrochloride*,



forms pearly scales, which do not melt at 310°. The *nitrate*, clusters of blunt prisms, the *sulphate*, $(\text{C}_5\text{H}_7\text{ON}_3)_2\text{H}_2\text{SO}_4\text{H}_2\text{O}$, large, flat prisms, and the *picrate*, yellow needles, decomposing above 265°, are also described.

J. C. C.

4-Keto-compounds of Pyrimines. AUGUST MICHAELIS and F. ENGELHARDT (*Ber.*, 1908, 41, 2668—2676).—The authors have prepared keto-compounds of the pyrimines by a similar method to that used for the preparation of the azo-derivatives of antipyrine and thiopyrine (Michaelis and Schlecht, *Abstr.*, 1906, i, 614), namely, by treating the corresponding ketopyrazolones or their chloropyrazolones with methyl sulphate. In the present paper, the 4-benzoyl derivatives of the pyrimines are described.

4-Benzoyl-1-phenyl-3-methyl-5-pyrazolone is best prepared by converting 1-phenyl-3-methyl-5-pyrazolone into the 5-benzoyl derivative, transforming the recrystallised substance into the 4:5-dibenzoyl derivative, and heating this with alcoholic potash according to Nef's method (*Abstr.*, 1892, 146). Nef (*loc. cit.*) describes 4-benzoyl-1-phenyl-3-methylpyrazolone as forming yellow needles, m. p. 86°, and Knorr (*Abstr.*, 1895, i, 396) gives m. p. 116—117°. The authors find that it exists in two isomeric modifications; the one separates from a mixture of light petroleum and chloroform in large, yellow, quadratic columns, m. p. 86°, and the other crystallises from alcohol in asbestos-like, white needles, m. p. 118°.

4-Benzoylantipyrine, $\begin{array}{c} \text{CMe:NMe} \\ | \text{O} < \\ \text{CBz:C} \end{array} \text{NPh}$, prepared from 4-benzoyl-1-

phenyl-3-methylpyrazolone and methyl sulphate, forms colourless, rhombic columns, m. p. 148°. Its aqueous solution gives a red coloration with ferric chloride. The *hydrochloride* crystallises in hexagonal tablets, m. p. 218°. The *phenylhydrazone*, $\text{C}_{11}\text{H}_{11}\text{ON}_2\cdot\text{CPh:N}\cdot\text{NPh}$, forms quadratic columns, m. p. 160°. The *hydrazones*,

$\text{C}_{11}\text{H}_{11}\text{ON}_2\cdot\text{CPh:N}\cdot\text{NH}_2$, crystallises in hexagonal prisms, m. p. 215°; with nitrobenzaldehyde it gives a yellow, insoluble compound, m. p. 350°; the *hydrochloride* of the hydrazone forms yellow, quadratic columns, m. p. 202°. The *oxime* of benzoylantipyrine, $\text{C}_{11}\text{H}_{11}\text{ON}_2\cdot\text{CPh:N}\cdot\text{OH}$, crystallises in long, hexagonal prisms, m. p. 197°.

4-*α*-Hydroxybenzylantipyrine, $\begin{array}{c} \text{CMe} = \text{N} \cdot \text{Me} \\ | \text{O} < \\ \text{C(CHPh}\cdot\text{OH):C} \end{array} \text{NPh}$, prepared

by reducing 4-benzoylantipyrine with sodium amalgam, crystallises in long, white, hexagonal prisms, m. p. 173°. It gives a strong antipyrine reaction with ferric chloride, and, on warming with dilute hydrochloric acid, furnishes benzylideneantipyrine hydrochloride (Knorr, *Abstr.*, 1884, 1378), which is found to have the formula $\text{C}_{20}\text{H}_{20}\text{O}_3\text{N}_4\cdot 2\text{HCl}\cdot 7\text{H}_2\text{O}$. Benzoylantipyrine and *α*-hydroxybenzylantipyrine have no striking poisonous effects when administered to dogs.

5-Chloro-4-benzoyl-1-phenyl-3-methylpyrazole methiodide, prepared by treating the pyrazole (Michaelis and Bender, *Abstr.*, 1903, i, 288) with methyl sulphate and subsequently adding potassium iodide to the aqueous solution, forms glistening, quadratic tablets, m. p. 166°. With sodium hydroxide, it yields 4-benzoylantipyrine, and with potassium

hydrogen sulphide it gives 4-benzylthiopyrine, $\begin{array}{c} \text{CMe:NMe} \\ | \text{S} < \\ \text{CBz:C} \end{array} \text{NPh}$,

crystallising in flat, quadratic prisms, m. p. 217°. The *phenylhydr-*

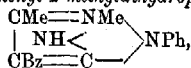
azone, $C_{11}H_{11}N_2S \cdot CPh \cdot N \cdot NPh$, crystallises in microscopic, yellowish-white needles, m. p. 280° , and the *methiodide* forms long, hexagonal prisms, m. p. 168° . When the latter is heated, it yields 4-benzoyl- ψ -thiopyrine (Michaelis and Lehmann; this vol., i, 691).

4-Benzoylanilopyrine, $\begin{array}{c} CMe=NMe \\ | \\ NPh < \text{---} C \text{---} \\ | \\ CBz \end{array} NPh$, prepared from 5-chloro-

4-benzoyl-1-phenyl-3-methylpyrazole methiodide and aniline, forms dark yellow, rhombic tablets, m. p. 159° ; the *phenylhydrazone*,

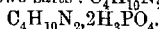
$C_{17}H_{16}H_3 \cdot CPh \cdot N \cdot NPh$, forms long, yellowish-white needles, m. p. 178° . The *methiodide* crystallises in large, colourless or pale rose-coloured, quadratic tablets, m. p. 172° , and, on heating, furnishes 4-benzoyl- ψ -anilopyrine (4-benzoyl-5-methylanilino-1-phenyl-3-methylpyrazole), $N \begin{array}{c} NPh \cdot C \cdot NMePh \\ | \\ CMe \cdot CBz \end{array}$, which forms hexagonal columns, m. p. 144.5° .

The *methiodide* of 5-amino-4-benzoyl-1-phenyl-3-methylpyrazole (Michaelis and Bender, *loc. cit.*) crystallises in glistening, quadratic tablets, m. p. 238° . The *methochloride* forms glistening, white leaflets, and with aqueous sodium hydroxide gives 4-benzoyliminopyrine (2:5-imino-4-benzoyl-1-phenyl-2-methyldihydropyrazole),

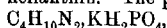


which crystallises in small, yellow needles, m. p. 155° . J. C. C.

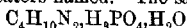
Piperazine Phosphates and Arsenates. A. ASTRUC and R. BRENTA (*Bull. Soc. chim.*, 1908, [iv], 3, 963—966).—Piperazine forms with phosphoric acid the two salts: $C_4H_{10}N_2 \cdot H_3PO_4 \cdot H_2O$ and



Of these, both are acid to phenolphthalein, and the first is alkaline and the second neutral to helianthin. The potassium salt,



is neutral to phenolphthalein and alkaline to helianthin. The two arsenates are similar in constitution to the phosphates, and show like behaviour with the indicators named. The salts of the type

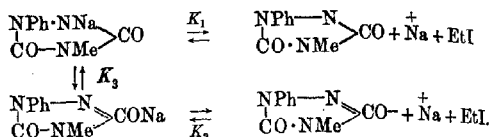


may be regarded as analogous with disodium hydrogen phosphate or arsenate, and those of the type $C_4H_{10}N_2 \cdot 2H_3PO_4$ may perhaps be regarded as salts of the acid, $2H_3PO_4$, mentioned by Giran (this vol., ii, 686).

T. A. H.

Urazoles XIV. Reactions between Tautomeric Acids and Salts with Diazomethane and Alkyl Haloids. SALAMON F. ACREE, J. M. JOHNSON, R. F. BRUNEL, G. H. SHADINGER, and SIDNEY NIEDLINGER (*Ber.*, 1908, 41, 3199—3236. Compare this vol., ii, 163).—1-Phenylurazole and its derivatives are moderately strong acids, and yield salts not markedly hydrolysed, the dissociation constants of which are easily determined. They also react easily with alkyl halides, and as no slow tautomeric changes occur and the isomeric ethers so obtained are stable, these compounds are very

suitable for an investigation of tautomeric phenomena. It has now been proved with practical certainty that both forms of the salts of 1-phenyl-4-methylurazole exist in solution, and that the alkyl halide reacts with the enolic as well as with the ketonic anion to form the O- and N-ethers.



If K_3 is the alkylation constant, x the velocity of formation of the N-ether, and x' that of the O-ether, then

$dx/dt = K(C_{\text{salt}} - x - x')(C_{\text{EtI}} - x - x')$; $d(x+x')/dt = K + K'(C_{\text{salt}} - x - x')(C_{\text{EtI}} - x - x')$, and $K/K' = x/x' = \text{N-ether/O-ether}$. The ratio between the quantities of the two stable alkyl or other derivatives from tautomeric substances must be independent of the time, and the ratio between the quantity of ethers and the alkylating reagent is dependent on three factors: (1) the relative reactivities of the tautomeric isomerides with the reagent; (2) the ratio of the isomerides if these exist in equilibrium, and (3) the velocity of transformation of one isomeride into the other as soon as the equilibrium is disturbed.

The hypotheses of Comstock, Wheeler, and Nef are discussed from this point of view, and considered to be erroneous. The theory (*loc. cit.*) has been confirmed and extended to all tautomeric phenomena. It would appear that the equilibrium is established between the molecules of the salt, and not the anions, as the addition of sodium iodide to solutions of ethyl iodide and sodium urazole does not cause a catalytic alteration in the ratio of O-ether to N-ether, although the nature of the urazole salt has a large effect. Thus ethyl iodide gives with the sodium, potassium, barium, and zinc salts of 1-phenyl-4-methylurazole in a 0.3*N* solution in 40% alcohol at 60°, velocity constants: 0.42, 0.36, 0.28, and 0.05. The sodium salt of 1-phenyl-2-methylurazole yields with methyl iodide, 98% of the N-ether and 2% of the O-ether, whereas the silver salt gives a 50% yield of both. Experiments are quoted to show that by using the potassium and silver salts of 1-phenyl-4-methylurazole with ethyl iodide at 22°, 60°, and 90°, constant values of the ratio of two ethers are obtained; thus indicating that the equilibrium in solution is not markedly affected by temperature.

The effect of solvent has also been studied; thus the silver salt with ethyl iodide at 60° gives the ratio of the two ethers: in 40% alcohol, 40:60; in absolute alcohol, 33:67; in acetone, 39.5:60.5; in water, 37:63, and in ether, 35:65.

When different alkyl iodides are employed, this ratio of N- to O-ether is altered. The silver salt of 1-phenyl-2-methylurazole reacts quickly with methyl iodide to form 50% of each, and with ethyl iodide only slowly to give 90% O- and 10% N-ether.

Concentration has practically no effect; thus the sodium salt of

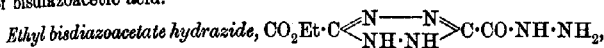
1-phenyl-4-methylurazole with ethyl iodide in 0.3, 0.15, 0.1, and 0.075*N* solutions gave the ratio 94 N- : 6 O-ether. W. R.

Action of Hydrazine Hydrate on Ethyl Bisdiazooacetate.

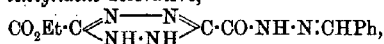
THEODOR CURTIUS and EUGEN RIMELE (*Ber.*, 1908, 41, 3108—3116).

—In continuation of the investigation of Curtius, Darapsky, and Bockmühl (this vol., i, 144), the behaviour of ethyl bisdiazooacetate towards hydrazine has been studied. It is found that the carbethoxy-groups of ethyl bisdiazooacetate are attacked by hydrazine hydrate, the first compound formed being ethyl bisdiazooacetate hydrazide, which is acted on by further quantities of hydrazine, yielding bisdiazooacetic acid dihydrazide. In this respect, ethyl bisdiazooacetate behaves like ethyl terephthalate; in both compounds, the carbethoxy-groups occupy 1:4-positions.

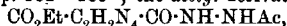
Ethyl bisdiazooacetate hydrazide is converted by nitrous acid into ethyl tetrazinedicarboxylate azoimide, and the dihydrazide into tetrazinedicarboxylic acid diazoimide. Ethyl bisdiazooacetate is most readily prepared by the action of diazomethane on an ethereal solution of bisdiazooacetic acid.



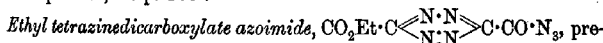
prepared by the action of hydrazine hydrate on ethyl bisdiazooacetate in alcoholic solution, crystallises in small, soft, pale yellow needles, m. p. 228—231° (decomp.); the *hydrochloride*, $\text{C}_8\text{H}_{10}\text{O}_3\text{N}_6 \cdot \text{HCl}$, crystallises in slender, golden-yellow needles, m. p. 212°. The corresponding *methyl ester*, $\text{C}_8\text{H}_8\text{O}_3\text{N}_6$, has m. p. 211°. The following derivatives of the ethyl ester are prepared by shaking its solution in dilute hydrochloric acid with the aldehyde or by heating the hydrazide with the ketone: the *benzylidene* derivative,



is obtained as a yellow, crystalline powder, decomposing at 233—234°; the *p-methoxybenzylidene* derivative, $\text{C}_{14}\text{H}_{16}\text{O}_3\text{N}_6$, sinters and melts at 237°; the *propylidene* derivative, $\text{CO}_2\text{Et} \cdot \text{C}_2\text{H}_5\text{N}_4 \cdot \text{CO} \cdot \text{NH} \cdot \text{N} \cdot \text{CMe}_3$, crystallises in slender, pale yellow needles, m. p. 115°; the *α-phenyl-ethylidene* derivative, $\text{C}_{14}\text{H}_{16}\text{O}_3\text{N}_6$, crystallises in small, glistening, pale yellow needles, m. p. 182—185°; the *acetyl* derivative,

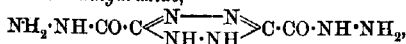


prepared by acting on the hydrazide with acetic anhydride, forms a yellow powder, m. p. 166°.



prepared by treating a solution of the hydrazide hydrochloride with sodium nitrite, crystallises in violet-red leaflets, decomposes when heated, and liberates 2 mols. of nitrogen when its alcoholic solution is boiled.

Bisdiazooacetic acid dihydrazide,



obtained by the action of a large excess of hydrazine hydrate on ethyl bisdiazooacetate, crystallises in golden-yellow spangles, m. p. 265—275°

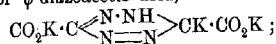
(decomp.); the *hydrochloride*, $C_6H_5O_2N_4 \cdot 2HCl$, and *dibenzylidene derivative*, $C_6H_5N_4(CO \cdot NH \cdot N \cdot CHPh)_2$, a yellow powder, m. p. above 290° , were prepared and analysed.

Hydrazine bisdiazooacetate, $C_2H_2N_4(CO_2 \cdot N_2H_5)_2$, prepared by adding hydrazine hydrate to an alcoholic solution of bisdiazooacetic acid, crystallises in small, yellow needles, m. p. $183-188^\circ$. *Ammonium hydrogen bisdiazooacetate*, $CO_2H \cdot C_2H_2N_4 \cdot CO_2NH_4$, prepared by adding the acid (1 mol.) to the neutral ammonium salt (1 mol.), crystallises in orange-red needles, m. p. 192° . In the same way are obtained: *sodium hydrogen bisdiazooacetate*, $C_2H_2O_4N_4Na \cdot H_2O$, yellow needles, and *potassium hydrogen bisdiazooacetate*, $C_2H_2O_4N_4K$. *Barium bisdiazooacetate*, $C_4H_2O_4N_4Ba \cdot 2H_2O$, is obtained as a yellow precipitate by adding barium chloride to an alcoholic solution of the acid. Attempts to prepare acid barium and silver salts were unsuccessful.

W. H. G.

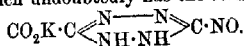
ψ -Diazooacetic Acid. ERNST MÜLLER (*Ber.*, 1908, 41, 3116--3139).

—A continuation of the investigations by Curtius, Darapsky, and Müller (compare Abstr., 1906, i, 939; 1907, i, 21, 262, 359, 450, 451). When ethyl diazoacetate is treated with a concentrated aqueous solution of potassium hydroxide, it is converted into a tripotassium salt of ψ -diazooacetic acid,



the formation of this salt is greatly accelerated by the presence of ethylalcohol. It is converted by dilute acetic acid into the dipotassium salt, $OO_2K \cdot C \begin{array}{c} \text{N} \cdot \text{NH} \\ \text{N} = \text{N} \end{array} > CH \cdot CO_2K$, which, with glacial acetic acid, yields

the potassium hydrogen salt, $CO_2K \cdot C \begin{array}{c} \text{N} \cdot \text{NH} \\ \text{N} = \text{N} \end{array} > CH \cdot CO_2H$. That the latter has this constitution follows from the fact that it yields a nitroso-derivative which undoubtedly has the constitution



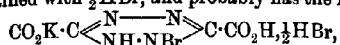
Potassium ψ -diazooacetate is hydrolysed by boiling acids, yielding glyoxylic acid (2 mols.), hydrazine (1 mol.), and nitrogen (1 mol.).

ψ -Diazooacetic acid has not yet been isolated, but an aqueous solution of the acid is obtained by treating the lead salt with hydrogen sulphide; the yellow solution thus obtained has a decided acid reaction, and gives the reaction characteristic of the ψ -series, namely, evolution of nitrogen and formation of hydrazine and glyoxylic acid when warmed with acids.

Unlike ψ -diazooacetamide, which is readily converted by a warm solution of potassium hydroxide into bisdiazooacetamide (compare Abstr., 1906, i, 939), potassium ψ -diazooacetate is converted only with difficulty into potassium bisdiazooacetate. The formation of potassium bisdiazooacetate from potassium diazoacetate observed by Hantzsch and Lehmann (Abstr., 1901, i, 678) must therefore take place through the intermediate ψ -diazooacetate.

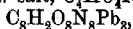
An aqueous solution of potassium ψ -diazooacetate is converted by bromine into potassium hydrogen bromodihydrotetrazinedicarboxylate;

it is always obtained with $\frac{1}{2}$ HBr, and probably has the formula



since it is also formed by the action of bromine on potassium bisdiazooacetate, and is reduced by hydrogen sulphide to bisdiazooacetic acid. All attempts to remove the $\frac{1}{2}$ HBr lead to the decomposition of the substance; thus it is converted by an aqueous solution of potassium acetate into potassium tetrazinedicarboxylate, and by sulphurous acid into tetrazinecarboxylic acid.

Tripotassium ψ -diazooacetate, $\text{C}_4\text{HO}_4\text{N}_4\text{K}_3 \cdot 2\text{H}_2\text{O}$, is obtained as a pale yellow precipitate, which becomes brownish-yellow when exposed to light; the *dipotassium* salt, $\text{C}_4\text{H}_2\text{O}_4\text{N}_4\text{K}_2 \cdot \text{H}_2\text{O}$, as a lemon-yellow precipitate; the *potassium hydrogen* salt, $\text{C}_4\text{H}_3\text{O}_4\text{N}_4\text{K} \cdot \text{H}_2\text{O}$, as a bright yellow precipitate; the *silver* salt, $\text{C}_4\text{HO}_4\text{N}_4\text{Ag}$, and *lead* salt,

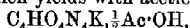


as yellow precipitates by adding silver nitrate or lead acetate to aqueous solutions of the dipotassium salt. *Dipotassium ammonium ψ -diazooacetate*, $\text{C}_4\text{HO}_4\text{N}_4\text{K}_2\text{NH}_4$, is obtained as hard, yellow leaflets by saturating a cold aqueous solution of the potassium salt with ammonia.

Potassium hydrogen bromodihydro-tetrazinedicarboxylate,



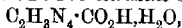
forms a brick-red powder; it is converted by an aqueous solution of potassium acetate into *potassium tetrazinedicarboxylate*, $\text{C}_4\text{O}_4\text{N}_4\text{K}_2$, a violet-red precipitate, which yields with acetic acid the salt,



obtained as an orange powder; the latter loses acetic acid at 90° , yielding *potassium hydrogen tetrazinedicarboxylate*, $\text{C}_4\text{HO}_4\text{N}_4\text{K}$, a carmine-red powder.

Ethyl tetrazinedicarboxylate, $\text{C}_2\text{N}_4(\text{CO}_2\text{Et})_2$, is prepared by the action of nitrous gases on an ethereal solution of ethyl bisdiazooacetate; it crystallises in long, purple prisms, m. p. $105\text{--}106^\circ$ (decomp.).

Potassium nitrosodihydro-tetrazinecarboxylate, $\text{CO}_2\text{K} \cdot \text{C}_2\text{H}_3\text{N}_4 \cdot \text{NO}$, is obtained by adding acetic acid to an aqueous solution of potassium ψ -diazooacetate and nitrite as a bright yellow precipitate, m. p. 170° (decomp.). It is converted by hydrogen sulphide into a *sulphur dioxide additive* compound of *potassium 1:2-dihydro-1:2:4:5-tetrazine-3-carboxylate*, obtained as a pale yellow, crystalline substance. A strong aqueous solution of this substance, when treated with sulphuric acid, yields *1:2-dihydro-1:2:4:5-tetrazine-3-carboxylic acid*,



which crystallises in glistening, yellow scales, m. p. $93\text{--}105^\circ$ (decomp.). When heated at $60\text{--}70^\circ$ for forty-five minutes, it is converted into 1-amino-3:4-triazole (compare Bülow, Abstr., 1906, i, 905; 1907, i, 99).

W. H. G.

So-called *Ethyl isoDiazooacetate*. THEODOR] CURTIUS, AUGUST DARAPSKY, and ERNST MÜLLER (*Ber.*, 1908, 41, 3140—3160).—Contrary to the statement of Hantzsch and Lehmann (*Abstr.*, 1901, i, 678), the compound described by these authors as ethyl

potassioisodiazooacetate, $\text{CO}_2\text{Et}\cdot\text{C}\begin{smallmatrix} \text{N} \\ \diagup \\ \text{N} \end{smallmatrix}\text{K}$, when treated with dilute acid liberates nitrogen; the solution is then found to contain ethyl glyoxylate (2 mols.) and hydrazine (1 mol.). If the solution is boiled for some time, the ethyl glyoxylate is hydrolysed, and the glyoxylic acid reacts with the hydrazine, yielding ammonia and oxalic acid. It is therefore evident that the compound is not ethyl potassioisodiazooacetate, since this substance when hydrolysed would yield only ethyl oxalate and hydrazine. The compound prepared according to the directions of Hantzsch and Lehmann is shown to have the composition $\text{CO}_2\text{Et}\cdot\text{C}\begin{smallmatrix} \text{N}\cdot\text{NH} \\ \diagup \quad \diagdown \\ \text{N}=\text{N} \end{smallmatrix}\text{CK}\cdot\text{CO}_2\text{Et}, \text{KOEt}$; that is, it is an additive product of potassium ethoxide with potassium diethyl ψ -diazooacetate (compare Müller, preceding abstract), which, when hydrolysed decomposes, thus: $\text{C}_2\text{H}_5\text{N}_4(\text{CO}_2\text{Et})_2 \xrightarrow{2\text{H}_2\text{O}} 2\text{CO}_2\text{Et}\cdot\text{CHO} + \text{N}_2\text{H}_4 + \text{N}_2$. An aqueous solution of this substance, contrary to the statements of Hantzsch and Lehmann (*loc. cit.*), yields with silver nitrate the silver salt, $\text{C}_8\text{H}_{11}\text{O}_4\text{N}_4\text{Ag}, \frac{1}{2}\text{Ag}_2\text{O}$, obtained as a greenish-black precipitate, and with mercuric chloride a dense, yellowish-brown precipitate of a complex mercury salt. The potassium salt is converted by an aqueous solution of potassium hydroxide (1:1) into potassium ψ -diazooacetate (compare Müller, *loc. cit.*).

Sodium diethyl ψ -diazooacetate, $\text{CO}_2\text{Et}\cdot\text{C}_2\text{HN}_2\cdot\text{Na}\cdot\text{CO}_2\text{Et}, \text{NaOEt}$, described by Hantzsch and Lehmann as ethyl sodioisodiazooacetate, $\text{CO}_2\text{Et}\cdot\text{CNaN}_2, \frac{1}{2}\text{H}_2\text{O}$, is best prepared by the action of sodium ethoxide, prepared by Brühl's method, on ethyl diazoacetate.

The substance which separates as an oil when an aqueous solution of either of the above salts is treated with dilute acids could not be purified, and all attempts to prepare derivatives were unsuccessful.

W. H. G.

Products of the Transformation of Ethyl Diazoacetate under the Influence of Alkalis. THEODOR CURTIUS, AUGUST DARAPSKY, and ERNST MÜLLER (*Ber.*, 1908, 41, 3161—3172).—A summary of the results obtained during the investigation on the behaviour of ethyl diazoacetate towards alkalis (compare Curtius, Darapsky, and Müller, *Abstr.*, 1906, i, 939; 1907, i, 21, 262, 359, 450, 451, and preceding abstract; Müller, preceding abstract).

W. H. G.

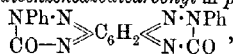
Ethylenediguanide. EMIL DITTLER (*Monatsh.*, 1908, 29, 645—652).—Whilst monoamines form additive compounds (the mono-substituted diguanides) with dicyanodiamide, Ziegelbauer observed (*Abstr.*, 1897, i, 142) that the action of *o*-phenylenediamine on dicyanodiamide leads to the formation of ammonia and *o*-phenylenediguanide, $\text{C}_6\text{H}_4\cdot\begin{smallmatrix} \text{NH}\cdot\text{C}(\text{NH}) \\ \diagup \quad \diagdown \\ \text{NH}\cdot\text{C}(\text{NH}) \end{smallmatrix}\text{NH}$. With the object of determining if this formation of a disubstituted diguanide is a general reaction of diamines, the author has studied the action of ethylenediamine on dicyanodiamide, and has found that here, also, the reaction leads to the formation of ammonia and a disubstituted diguanide.

Ethylenediguanide, $C_2H_4 \begin{smallmatrix} < NH \cdot C(NH) \\ < NH \cdot C(NH) \end{smallmatrix} > NH$, formed by the action of ethylenediamine on dicyanodiamide at 80—150°, is isolated as the sulphate of the copper derivative, $(C_4H_8N_5)_2Cu, H_2SO_4, 3H_2O$, which separates in rose-red, microscopic needles, and dissolves sparingly in water, but readily in aqueous alkalis, forming red solutions. The *hydrogen sulphate*, $C_4H_8N_5, H_2SO_4$, forms hygroscopic, white needles, and gives characteristic reactions with nickel, cobalt, gold, and platinum salts, and with chromic, picric, and picrolonic acids. The *sulphate*, $(C_4H_8N_5)_2, H_2SO_4$, *hydrogen chloride*, $C_4H_8N_5, 2HCl$, *chromate*, $(C_4H_8N_5)_2, H_2CrO_4$, and *picrolonate*, $C_4H_8N_5, C_{10}H_8O_5N_4$, are described. The free base, liberated from the sulphate by cold baryta water, is obtained as a syrup, which decomposes above 150°, yielding complicated products. The action of boiling baryta water on the sulphate, on the other hand, leads to the formation of ethylenediamine. G. Y.

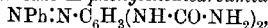
Aromatic Monatomic Cyanamides. PAUL PIERRON (*Ann. Chim. Phys.*, 1908, [viii], 15, 145—278).—Mainly a résumé of work already published (Abstr., 1905, i, 125; 1906, i, 772; 1907, i, 121); the following compounds are described for the first time: *ψ-cumylcyanamide*, $C_8H_7Me_3 \cdot NH \cdot CN$, colourless prisms, m. p. 126°; the *carbamide* and the *benzoyl* derivatives melt at 237° and 94° respectively; *p*-iodophenylcyanamide, $C_6H_4I \cdot NH \cdot CN$, m. p. 110°, and the *carbamide*, m. p. 244°; *benzeneazo-m-ethoxycyananilide*, $CN \cdot NH \cdot C_6H_3(OEt) \cdot N : NPh$, m. p. 81—82°; and the following corrections in melting points are recorded: the benzoyl derivative of benzeneazo-*p*-cyananilide has m. p. 191°, and not 161°; benzeneazo-*m*-tolylcarbamide has m. p. 186°, and not 152°; and benzeneazo-*p*-cyano-*o*-ethoxyanilide has m. p. 120°, and not 121° (compare Abstr., 1906, i, 772).

With the view of preparing the corresponding dicyanamides and their derivatives, the author has examined the behaviour of the phenylenediamines with cyanogen bromide.

m-Phenylenediamine yields *m*-phenylenedicyanamide, $C_6H_4(NH \cdot CN)_2$, m. p. 205—207°. When heated rapidly, it condenses with diazobenzene chloride to yield the following products: (1) *benzeneazo-m-phenylenedicyanamide*, $C_6H_3(CN \cdot NH)_2(N : NPh)_2$, m. p. 250—252°; (2) *m-bis-keto-2-phenylbenzotriazine* (*dibenzeneazodicarbonyl-m-phenylenediamine*)



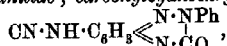
p. p. 310°; (3) *benzeneazo-m-phenylenedicarbamide*,



m. p. 238—240°; (4) *imino-m-cyanamido-2-phenylbenzotriazine benzeneazoaminocarbonylaminophenylcyanamide*; *iminocarbonylcyanohyrsoidine*, $CN \cdot NH \cdot C_6H_3 \rightleftharpoons N \cdot NPh$ m. p. 200° when heated rapidly, or 290° when gradually heated; the *hydrochloride* has m. p. 230°.

m-Cyanaminophenylcarbamide, $CN \cdot NH \cdot C_6H_4 \cdot NH \cdot CO \cdot NH_2$, m. p. 10°, has a pronounced sweet taste, and condenses with diazobenzene chloride to form *keto-m-cyanamido-2-phenylbenzotriazine* (*benzeneazo-*

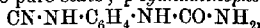
carbonyl-m-amidocyananilide; carbonylcyanochrysoidine),



m. p. 258°; the corresponding carbamide has m. p. 300°, and, on reduction, it yields keto-m-cyanamido-2-phenyldihydrobenzotriazine (dihydrocarbonylcyanochrysoidine), $\text{CN}\cdot\text{NH}\cdot\text{C}_6\text{H}_5 \begin{smallmatrix} \text{NH}\cdot\text{NPh} \\ \text{NH}\cdot\text{CO} \end{smallmatrix}$, m. p. 210°.

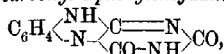
m-Cyanoaminoacetanilide, $\text{CN}\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{NHAc}$, m. p. 205–207°, condenses with diazobenzene chloride to form benzeneazoacetamidocyananilide (acetylcyanochrysoidine), $\text{CN}\cdot\text{NH}\cdot\text{C}_6\text{H}_4(\text{NAc})\cdot\text{N}\cdot\text{NPh}$, m. p. 246°; cyanochrysoidine, $\text{CN}\cdot\text{NH}\cdot\text{C}_6\text{H}_4(\text{NH})\cdot\text{N}\cdot\text{NPh}$, has m. p. 186°, and the benzoyl derivative has m. p. 233°.

In the case of p-phenylenediamine, the dicyanamide derivative could not be isolated in the pure state; p-cyanaminophenylcarbamide,



has m. p. 255°, and does not form a stable compound with diazobenzene chloride.

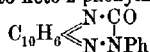
o-Phenylenediamine does not form cyanamide derivatives, but yields o-phenyleneguanidine, $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{NH} \\ \text{NH} \end{smallmatrix} > \text{C}\cdot\text{NH}$, m. p. 222°, when treated with cyanogen bromide; the benzoyl derivative has m. p. 236°; it is converted into iminodicarbonyl-o-phenyleneguanidine,



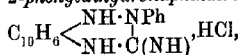
by the further action of cyanogen bromide, and the same compound is obtained by the action of cyanogen bromide on o-phenylenediamine in the presence of sodium hydrogen carbonate, or by the condensation of o-phenyleneguanidine with biuret.

β-Naphthylcyanamide condenses with diazobenzene chloride to form imino-2-phenylnaphthatriazine (benzeneazoiminocarbonyl-β-naphthylamine), $\text{C}_{10}\text{H}_6 \begin{smallmatrix} \text{N}\cdot\text{NPh} \\ \text{N}\cdot\text{C}(\text{NH}) \end{smallmatrix}$, m. p. 160°, and the same compound is

obtained by the action of cyanogen bromide on benzeneazo-β-naphthylamine; the hydrochloride, m. p. 230°, and the dichromate have been prepared. When the base is treated with acids, alkalis, or benzoyl chloride, it is converted into keto-2-phenylnaphthatriazine,



(compare Goldschmidt and Rosell, Abstr., 1890, 614; Busch, Abstr., 1900, i, 59), and, on reduction with stannous chloride, it yields the hydrochloride of imino-2-phenyldihydronaphthatriazine,



m. p. 242–245°.

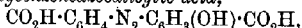
M. A. W.

Azo-compounds of Salicylic Acid. EUGÈNE GRANDMOUGIN and JULIEN GUISAN (*Chem. Zentr.*, 1908, ii, 310–311; from *Rev. gén. Matières Colorantes*, 1908, 12, 129–137. Compare Abstr., 1907, i,

987).—When aminosalicilic acid and *p*-phenylenediamine are reduced by means of sodium hydrogen sulphite in hot aqueous solution, *p*-nitrobenzeneazosalicylic acid, m. p. 256°, is formed.

m-Tolueneazosalicylic acid is a yellow, crystalline substance, m. p. 208°; acetyl compound, m. p. 155°. *p*-Tolueneazosalicylic acid forms yellowish-brown needles, m. p. 214°; the sodium salt is known as flavazole, and the acetyl compound, $C_{16}H_{14}O_3N_2$, pale yellow crystals, m. p. 159.5°. *m*-Nitrotolueneazosalicylic acid, $C_{14}H_{11}O_5N_3$, prepared from diazotised *m*-nitro-*p*-toluidine and salicylic acid in alkaline solution, forms yellow needles, m. p. 242°; the acetyl compound, $C_{16}H_{13}O_6N_3$, large, yellow crystals, m. p. 171°.

o-Nitrotolueneazosalicylic acid, prepared in like manner, crystallises in brown needles, m. p. 210°, which, when reduced with hydrogen sulphite, form a triazole compound; the acetyl compound crystallises in brownish-yellow needles, m. p. 180°. *p*-Acetylaminobenzeneazosalicylic acid, $C_{15}H_{13}O_4N_3$, forms a brownish-yellow, crystalline powder, m. p. 245° (decomp.); the diacetyl compound, yellow needles, m. p. 200° (decomp.). When this is treated with strong sulphuric acid, *p*-aminobenzeneazosalicylic acid, $NH_2 \cdot C_6H_4 \cdot N_2 \cdot C_6H_3(OH) \cdot CO_2H$, is produced, which forms a grey, crystalline powder, m. p. 230° (decomp.), and gives with carbonyl chloride the diaminodiphenyl derivative of carbamide, $CO[NH \cdot C_6H_4 \cdot N_2 \cdot C_6H_3(OH) \cdot CO_2H]_2$. *p*-Hydroxybenzeneazosalicylic acid, $C_{13}H_{10}O_4N_2$, prepared by the diazo-reaction from *p*-aminobenzeneazosalicylic acid, or from *p*-aminophenol and salicylic acid, or from aminosalicilic acid and phenol, crystallises in brown needles, m. p. 236°. *p*-Ethoxybenzeneazosalicylic acid, $C_{15}H_{14}O_4N_2$, prepared from diazotised phenetidine and salicylic acid, is a green, crystalline powder, m. p. 208°; the acetyl compound, yellow crystals, m. p. 158.5°. *m*-Carboxybenzeneazosalicylic acid,



prepared from *m*-aminobenzoic acid, has m. p. about 280° (decomp.). *o*-Carboxybenzeneazosalicylic acid, from anthranilic acid, forms yellow crystals, m. p. 219° (with sublimation). *m*-Sulphobenzeneazosalicylic acid, prepared from metanilic acid, gives a crystalline sodium salt of the composition $Na \cdot C_9H_6O_5N_2S \cdot 3H_2O$. When diazotised nitroaniline and diazotised 2-naphthylamine-7-sulphonic acid act on salicylic acid, some tris-azo-compound and phenol-bis-azo-compound are found together with the bis-azo-derivative.

J. V. E.

Aromatic and Aliphatic *iso*Diazo-compounds (*iso*Azotates) from Hydrazines. JOHANNES THIELE (*Ber.*, 1908, 41, 2806–2811).—When phenylhydrazine is treated with an alkyl nitrite and alkali, it is smoothly converted into the corresponding metallic salt of *iso*-diazobenzene, nitrogen monoxide being formed at the same time. In the well-known production of diazonium salts from phenylhydrazine salts and nitrous acid and from nitrosophenylhydrazine, the author has been able to show that nitrogen monoxide is formed here also, and he considers that the process involves the intermediate formation of dinitrosohydrazines, $R \cdot N(NO) \cdot NH \cdot NO$, or the alkali salts of their tautomerides. It is probable, also, that in the interation of hydrazine

and nitrous acid (Dennstedt and Göhlich, Abstr., 1898, ii, 425), dinitrosophydrazine is formed, which, with the possible intermediate formation of a compound $N_2 \cdot NO$, may decompose according to the equation: $NO \cdot NH \cdot NH \cdot NO = N_2O + N_2 + H_2O$. Further, azoimide and nitrous acid decompose, thus: $N_2H + HNO_2 = N_2 + N_2O + H_2O$, so that for each molecule of nitrogen monoxide formed in the above reaction one molecule of azoimide is lost.

Nitrosophenylhydrazine forms salts with bases, and, on heating its solutions in indifferent solvents, it decomposes into aniline and nitrogen monoxide; moreover, it does not form nitrosophydrazones with acetaldehyde or benzaldehyde, and it is suggested that its constitution may be represented as $C_6H_5 \cdot NH \cdot NH \cdot NO$, or the tautomeric expression.

Nitrosobenzylhydrazine gives the first two reactions, but gives the normal hydrazone with benzaldehyde, so that the symmetrical formula $CH_2Ph \cdot NH \cdot NH \cdot NO$ cannot be assigned to it. Benzylhydrazine also gives the corresponding isodiazotate, and, as this may be regarded as belonging to the aliphatic series, it is termed *sodium benzylisodiazotate*, $C_6H_5 \cdot CH_2 \cdot N \cdot N \cdot ONa$. This compound, which forms white needles, resembles sodium benzenedisodiazotate in its reactions, except that it cannot be converted into a diazonium salt and does not yield azo-dyes; with acids, nitrogen is evolved.

Sodium benzenedisodiazotate, prepared by adding ethyl nitrite mixed with ether to a cold mixture of phenylhydrazine, sodium methoxide, and ether, gives, in aqueous solution, with ferric chloride a brown, with copper sulphate a blackish-violet, and with mercuric nitrate a white precipitate. Sodium *p*-nitrobenzenedisodiazotate and sodium benzylisodiazotate give similar reactions.

An aqueous solution of nitrosophenylhydrazine gives with ferric chloride a transient violet coloration, with copper acetate a precipitate followed by reduction, and with mercuric nitrate a yellow precipitate sparingly soluble in dilute nitric acid. The *sodium* salt, $C_6H_5 \cdot N_2HONa$, obtained by adding sodium methoxide to the ethereal solution, is white; with water, nitrosophenylhydrazine is regenerated.

Nitrosobenzylhydrazine gives with copper acetate a pale precipitate soluble in dilute acids, which reddens on warming. With mercuric nitrate, a yellow precipitate, soon turning white, is produced, which is sparingly soluble in nitric acid. The *sodium* salt, $C_6H_5N_2 \cdot ONa$, resembles that of nitrosophenylhydrazine. J. C. C.

Hydrolysis of Legumelin from the Pea (*Pisum sativum*).
THOMAS B. OSBORNE and FREDERICK W. HEYL (*J. Biol. Chem.*, 1908, 5, 197—206).—Legumelin is the albumin which occurs in many leguminous seeds. The following table compares the percentage of hydrolytic products in legumelin and leucosin, the albumin of wheat:

Legumelin. Leucosin.		Legumelin. Leucosin.	
Glycine	0.50 0.94	Glutamic acid ...	12.96 6.73
Alanine	0.92 4.45	Tyrosine	1.56 3.34
Valine	0.69 0.18	Arginine	5.45 5.94
Leucine	9.63 11.34	Histidine	2.27 2.83
Proline	3.96 3.18	Lysine	3.03 2.75
Phenylalanine ...	4.79 3.83	Ammonia	1.26 1.41
Aspartic acid	4.11 3.35		
		Total	51.15 50.53

Tryptophan is present in both; serine and cystine were not estimated.

W. D. H.

Hydrolysis of Vicilin from the Pea (*Pisum sativum*). THOMAS B. OSBORNE and FREDERICK W. HEYL (*J. Biol. Chem.*, 1908, 5, 187—196).—Vicilin is a globulin occurring in the pea, horse-bean, and lentil. It differs from legumin in containing more carbon, less nitrogen, and much less sulphur. It contains the smallest proportion of sulphur yet found in any protein (0.1—0.2%). The following table compares the percentage of hydrolytic products of the two proteins:

	Vicilin.	Legumin.		Vicilin.	Legumin.
Glycine	0.0	0.38	Cystine	not determined	
Alanine	0.5	2.08	Oxyproline	"	"
Valine	0.15	?	Tyrosine	2.38	1.55
Leucine	9.38	8.00	Arginine	8.91	11.71
Proline	4.06	3.22	Histidine	2.17	1.69
Phenylalanine ...	3.82	3.75	Lysine	5.49	4.98
Aspartic acid	5.30	5.30	Ammonia	2.03	2.05
Glutamic acid	21.34	16.97	Tryptophan	present	present
Serine	?	0.53			
			Total	65.44	62.22

W. D. H.

Applicability of the Laws of Amphoteric Electrolytes to Serum Globulin. T. BRAILSFORD ROBERTSON (*J. Biol. Chem.*, 1908, 5, 155—162).—Polemical against H. Lundén (*ibid.*, 267).

W. D. H.

The Swelling of Fibrin. MARTIN H. FISCHER (*Pflüger's Archiv*, 1908, 125, 99—110. Compare Abstr., 1907, i, 1095).—Fibrin swells more in acids than in distilled water; various acids produce this effect better than others; for instance, hydrochloric acid is the most and sulphuric acid the least effective of those investigated. The amount of swelling is dependent on the concentration of the acids. Alkalis also cause fibrin to swell in various degrees in proportion to the concentration. Beyond a certain point in the case both of acid and alkali, the swelling decreases. Alkalis produce more swelling than acids of the same strength. The addition of salts lessens the swelling, as also do non-electrolytes (urea, sugar, &c.). The taking up and giving out of water is a reversible phenomenon.

W. D. H.

Solubility of Zein in Different Solvents. GINO GALEOTTI and J. GIAMPALMO (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 118—126).—Zein (a protein which occurs in maize) is insoluble in water and in alcohol, but is readily soluble in a mixture of them. The maximum solubility is observed with 60% alcohol. The solutions

in alcohol stronger than 45% are clear and mobile, those in weaker alcohols are turbid and viscous. The addition of xylene, ether, or light petroleum does not precipitate zein from these solutions unless enough is added to form a second liquid phase. The solubility curve for mixtures of chloroform and 85% alcohol is found to possess two maxima, one about 16% and the other about 54% of chloroform. The solubility of zein in mixtures of water, alcohol, and acetone is very thoroughly studied, and the results represented by a diagram. The mixtures containing acetone dissolve less zein than the corresponding mixture of water and alcohol. The two series of solutions (clear and easily filtered—turbid and not readily filtered) are also found in the acetone mixtures. Zein is insoluble in all the pure solvents tried.

T. E.

Another Method of Measuring Large Molecular Masses. WILLIAM SUTHERLAND (*Phil. Mag.*, 1908, [vi], 16, 497—512).—From a relationship, previously deduced by the author, between the molecular conductivity, the concentration, the viscosities of the solvent and solution, the mass of the atom of hydrogen, the dielectric capacity of the solvent, and the dissociating force which causes complete ionisation, the author has deduced the molecular masses of globulin and casein. The globulin ions produced by hydrochloric acid in aqueous solutions are found to be bivalent, and their molecular mass about 10,000. The molecular mass of the casein ion is given as 2605. On the assumption that the casein ion is bivalent, this result agrees freely well with the value 1135 obtained by Laqueur and Sackur for its equivalent weight.

H. M. D.

Influence of Temperature on the Solubility of Casein in Alkaline Solutions. T. BRAILSFORD ROBERTSON (*J. Biol. Chem.*, 1908, 5, 147—154).—The solubility of casein in alkaline solutions is increased at temperatures over 40°. This fact is not in harmony with the view that heat increases the degree of hydrolytic dissociation of solutions of the caseinates. Osborne showed that heating increases the alkalinity and electrical conductivity of caseinate solutions; it is, therefore, suggested that the influence of heat on proteins consists, among other things, of shifting equilibria of the type:



in the direction of higher complexes, and that heat coagulation is a result of repeated condensations of this type. The solubility of casein in solutions of various concentrations of potassium hydroxide, lithium hydroxide, and calcium hydroxide at various temperatures is given.

W. D. H.

Products of Hydrolysis of Casein. ZDENKO H. SEBAUF (*Monatsh.*, 1908, 29, 791—798. Compare Abderhalden and Funk, *Abstr.*, 1907, i, 1095).—In the products of hydrolysis of casein with hydrochloric acid, three substances occur, which have the composition of leucylvalylimide. The one is fairly strongly levorotatory, the second is weakly rotatory, and the third is practically inactive. It is not yet certain whether these compounds are pure or mixtures.

The ether-alcoholic extract of the hydrolysed casein, on distillation, left a residue which was esterified and distilled in a vacuum; three fractions were collected up to 115° , and the residue on extraction with alcohol gave a substance, $C_{11}H_{20}O_2N_2$, sintering at 283° , m. p. 289° , $[\alpha]_D - 53.0^{\circ}$ (Abderhalden and Funk give $[\alpha]_D - 44.27^{\circ}$).

The mother liquor from this yielded a substance, $C_{11}H_{20}O_2N_2$, forming needle-shaped crystals, m. p. 250° , $[\alpha]_D - 17.6^{\circ}$. The alcoholic extract from which the first substance was obtained yielded, with light petroleum, a third substance, $C_{12}H_{20}O_2N_2$, forming crystals, m. p. 275° . The mother liquor from this gave leucine, and amines of the composition of butyl- and amyl-amine. After separating the leucine, the solution furnished a basic aurichloride, $C_6H_{12}NOCl_3Au$, m. p. 195° (decomp.), the corresponding hydrochloride of which crystallises only in a vacuum, and a platinichloride, $(C_6H_{13}N)_2H_2PtCl_6$, large, yellow leaves, m. p. 210° (decomp.); the corresponding hydrochloride crystallises more readily than that prepared from the aurichloride, and both give the carbamide reaction.

J. C. C.

Formation of Thymine by Heating Intestinal Nucleic Acid. KATSUJI INOUE (*Zeitsch. physiol. Chem.*, 1908, 57, 313).—If nucleic acid (from the intestines) is burnt, a white material collects on the walls of the tube. This was collected and identified as thymine.

W. D. H.

Pyrimidines. X. Action of Diazobenzenesulphonic Acid on Thymine, Uracil, and Cytosine. TREAT B. JOHNSON and SAMUEL H. CLAPP (*J. Biol. Chem.*, 1908, 5, 163—172).—Thymine, uracil, and cytosine all give, in the presence of alkali, a red solution with diazobenzenesulphonic acid. The colour given by thymine is most intense. Substitution in position 3 in the pyrimidine ring prevents the formation of a red colour, and the absence of this colour reaction in nucleic acids (Burian) indicates that thymine, and probably also cytosine and uracil, are linked in nucleic acids at position 3. Whether the pyrimidines are linked to phosphorus, a carbohydrate complex or otherwise, is not yet settled.

W. D. H.

Cleavage Products of Nucleic Acid. PHEBUS A. LEVENE (*Zeitsch. physiol. Chem.*, 1908, 57, 154—156; HERMANN STEUDEL, *ibid.*, 157—160).—Polemical between the two authors, mainly in reference to priority.

W. D. H.

Inosic Acid. PHEBUS A. LEVENE and WALTER A. JACOBS (*Ber.*, 1908, 41, 2703—2707).—When hydrolysed with 5% sulphuric acid and the products isolated by means of their barium salts, inosic acid furnishes a complex phosphoric acid, of which two barium salts were analysed. The one, $(C_5H_8O_8P)_2Ba_3$, is amorphous, has $[\alpha]_D^{20} + 4.4^{\circ}$, and reduces Fehling's solution on heating, and the other, $C_5H_7O_8PBa, 5H_2O$ or $C_5H_8O_8PBa, 5H_2O$, forms bundles of needles and readily reduces Fehling's solution. By hydrolysis of inosic acid with 5% potassium hydroxide, 30% of the original phosphoric acid is eliminated. The conclusion is drawn that in inosic acid the carbohydrate forms a

glucophosphoric acid with the phosphoric acid, and that the base is combined with the aldehyde group of the sugar. The authors are unable to confirm the observations of Neuberg and Brahn (Abstr., 1907, i, 1097), or of Bauer (Abstr., 1907, i, 1098), as to the nature of the sugar.

J. C. C.

Hydrolysis of Witte's Peptone. PHOEBUS A. LEVENE and D. D. VAN SLYKE (*Biochem. Zeitsch.*, 1908, 13, 440—457).—The peptone was hydrolysed by hydrochloric acid. The following yields of amino-acids were obtained (in parts per cent. of the peptone), the corresponding yields from fibrin being given in brackets: tyrosine, 3.25 (3.3); glycine, 0.78 (2.2); alanine, 2.83 (3.1); valine and leucine, 14.70 (13.0); phenylalanine, 2.60 (1.2); glutamic acid, 8.24 (6.6); aspartic acid, 1.70 (1.7); proline, 4.56 (2.4); serine, 1.18; histidine, 0.75; lysine, 2.71; arginine, 1.48. Total yield of amino-acids, 47.78%.

S. B. S.

Polypeptides Containing Tryptophan. EMIL ABERHALDEN and LOUIS BAUMANN (*Ber.*, 1908, 41, 2857—2860).—*Iodoacetyl-L-tryptophan*, prepared by coupling the components in alkaline solution, crystallises in needles, m. p. 175—176° (decomp.), and has $[\alpha]_D^{20} + 31.3^\circ$. *Chloroacetyl-L-tryptophan* has $[\alpha]_D^{20} + 32.9$, not -32.9° as previously stated in error (Abstr., 1907, i, 652). *Methyl-dl- α -iodopropionyl-L-tryptophan* crystallises in needles aggregated in rosettes, m. p. 145—146°. *dl-Alanyl-L-tryptophan anhydride*, prepared by the action of methyl-alcoholic ammonia on the foregoing, crystallises in minute needles, m. p. 290° (corr.), $[\alpha]_D^{20} + 87^\circ$.

E. F. A.

Plastein. PHOEBUS A. LEVENE and D. D. VAN SLYKE (*Biochem. Zeitsch.*, 1908, 13, 458—474).—The plastein was made by treatment of a 20% solution of Witte's peptone in 0.2% hydrochloric acid. It was submitted to hydrolysis, and the following percentages of amino-acids were obtained, the corresponding numbers for fibrin being given in brackets: tyrosine, 3.03 (3.1); glycine, 0.50 (2.2); alanine (3.1); valine and leucine, 15.59 (13.0); phenylalanine, 1.0 (1.2); glutamic acid, 10.02 (6.8); aspartic acid, 2.15 (1.7); proline, 2.55 (2.4); histidine, 0.43; arginine, 2.06; lysine, 1.42. Total yield of amino-acid, 38.75%. The numbers indicate that plastein is a complex protein, not far removed in composition from fibrin. It is not possible to determine, however, whether the plastein is a synthetic product, or a coagulated form of one of the fibrin albumoses.

S. B. S.

Organic Chemistry.

A New Reaction of Petroleum. ETTORE MOLINARI and P. FENAROLI (*Ber.*, 1908, 41, 3704—3707).—It has been found that Russian petroleum yields ozonides by the action of ozonised oxygen at 8—10°. An oil, b. p. 135—290°, gave 12% of ozonide, whereas one boiling at 295—300° gave 32%. The ozonide forms a white or pale yellowish-red powder soluble in ether, chloroform, or benzene. It can be kept for several days at low temperatures, but decomposes rapidly at 20°, and at 45—50° yields a soft, red substance which is converted into a brown resin at 150°.

The analyses of the ozonide agree with the conclusion that it is derived from a naphthene, $C_{17}H_{26}$, containing two ethylene linkings.

A Roumanian oil gave a similar ozonide, whereas an Italian oil, b. p. 140—265°, gave a small amount of an ozonide, probably derived from a hydrocarbon, $C_{15}H_{16}$.

The ozone also produces a certain amount of oxidation, and the residue left after the removal of the ozonide is still optically active, indicating that this activity cannot be due to unaltered cholesterol in the original oil.

J. J. S.

Preparation of Acetylene Di- and Tetra-chlorides. JOHANN H. LIDHOLM (D.R.-P. 201705).—A mixture of 2 vols. of chlorine and 1 vol. of acetylene, diluted with 10% of carbon dioxide, reacts quite quietly to furnish acetylene tetrachloride with about 10% of acetylene dichloride when exposed to radium emanations or to the light from a quartz-mercury lamp fed with a current of 3 to 5 amperes and 60—75 volts.

G. T. M.

Constitution of Copper Acetylde. JOHANNES SCHEIBER [with R. FLEBBE] (*Ber.*, 1908, 41, 3816—3828).—Makowka has assigned the constitution



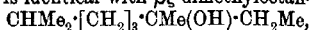
to the copper acetylene compound obtained by precipitation of a cuprous salt by acetylene (this vol., 328). It is now shown that the freshly-prepared compound can be converted into a dehydrated form, C_2Cu_2 , by heating for two hours at 100° over calcium chloride (compare Keiser, *Abstr.*, 1892, 1416). This result is not in accordance with the aldehyde formula. Further, no acetaldehyde can be detected when the compound is dissolved in hydrochloric acid, whereas trichloromercuriacetaldehyde yields the aldehyde, but no acetylene, under corresponding conditions (Biltz and Mumm, *Abstr.*, 1905, i, 2). It would also appear that the water is firmly held in the molecule, as after drying in a desiccator for twenty-eight days it still has $1H_2O$. Inasmuch as the compound is amorphous, it is unlikely that the compound contains water of crystallisation, and of two suggested formulæ, $\begin{array}{c} \text{Cu} \cdot \text{C} \\ | \\ \text{Cu} \cdot \text{C} \end{array} \cdot \frac{1}{2} H_2O$ and $CH:CCu \cdot CuOH$, preference

is given to the latter.

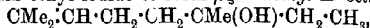
The carbonaceous residue left after dissolution of the acetylide in hydrochloric acid is not an impurity, but is due to secondary changes. Analysis of one preparation led to the formula $(C_{11}H_8O_4)_x$, but it is uncertain whether it is of definite composition.

W. R.

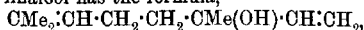
Hydrogenation of Linalool by means of Nickel and Hydrogen. Synthesis of Tetrahydrolinalool ($\beta\zeta$ -Dimethyloctan- ζ -ol). C. J. ENKLAAR (*Rec. trav. chim.*, 1908, 27, 411—417. Compare this vol., i, 664).—When linalool is directly hydrogenated in the presence of nickel at 130—140°, $\beta\zeta$ -dimethyloctane (compare Willstätter and Meyer, this vol., i, 383) is obtained together with the saturated alcohol, tetrahydrolinalool, $C_{10}H_{22}O$, b. p. 88—88.5°/14 mm., D_4^{25} 0.8360, n_D^{25} 1.4388, which is identical with $\beta\zeta$ -dimethyloctan- ζ -ol,



prepared synthetically from methylheptenone (Barbier and Bouveault, Abstr., 1896, i, 445; Verley, Abstr., 1898, i, 557; Leser, Abstr., 1898, i, 512; Tiemann, Abstr., 1898, i, 374; Ipatieff, Abstr., 1901, i, 256) by the following series of reactions: methylheptenone reacts with magnesium ethyl iodide to form $\beta\zeta$ -dimethyl- Δ^8 -octene- ζ -ol,



b. p. 94—94.5°, D_4^{25} 0.8695, n_D^{25} 1.4569, which, on direct hydrogenation in the presence of nickel, yields $\beta\zeta$ -dimethyloctan- ζ -ol; it follows therefore that linalool has the formula,



ascribed to it by Tiemann and Semmler (Abstr., 1895, i, 646).

M. A. W.

α -Methylallyl Alcohol (Δ^2 -Butene- γ -ol). ALFRED WOHL and M. S. LOSANITSCH (*Ber.*, 1908, 41, 3621—3622. Compare Reif, this vol., i, 847).—The action of magnesium methyl iodide on acraldehyde in ethereal solution leads to the formation of α -methylallyl alcohol, $CH_2 \cdot CH \cdot CHMe \cdot OH$, which is obtained as a viscid, colourless oil, b. p. 96—97°/756 mm. or 28—31°/35 mm., does not reduce ammoniacal silver or Fehling's solution, and when treated with permanganate is only partly oxidised, evolving carbon dioxide. Neither by this reaction nor by oxidation of the unsaturated alcohol with chromic acid and very dilute sulphuric acid can the corresponding ketone be obtained.

G. Y.

Complex Compounds of Multivalent Alcohols. A Class of Cyclic Complex Metallic Salts. ADOLF GRÜN and F. BOCKSCH (*Ber.*, 1908, 41, 3465—3478).— $\alpha\beta$ -Glycols and glycerol show a marked tendency to combine with metallic salts. Such compounds are regarded as having a cyclic structure, and are classed with the saccharates formed by sugars with metallic oxides. Ethylene glycol displaces water from the hydrates of copper, cobalt, nickel, and chromium salts, $C_2H_4(OH)_2$ replacing $2H_2O$.

Diethyleneglycolcuprosulphate, $CuSO_4 \cdot 2C_2H_4(OH)_2 \cdot 2H_2O$, prepared by warming the constituents together on the water-bath, crystallises in minute, bright blue needles, which melt to a clear blue liquid. On warming at 60°, it loses 25% of its weight, but still contains 11.3% of water. On prolonged heating at 125°, copper sulphate alone remains.

Triethyleneglycolcobaltosulphate, $\left[\text{Co} \left(\begin{smallmatrix} \text{CH}_2\cdot\text{OH} \\ \text{CH}_2\cdot\text{OH} \end{smallmatrix} \right)_3 \right] \text{SO}_4\cdot\text{H}_2\text{O}$, is best

obtained by heating the constituents at the water-bath temperature, diluting the saturated, purplish-red solution with a little alcohol after cooling, and adding acetone until the solution is decolorised. A red oil is precipitated, which forms minute, red, hygroscopic crystals on drying over sulphuric acid. It decomposes in aqueous solution, or on heating to 80° .

Glycerol monomethyl ether (α -monomethylin), prepared by the action of sodium methoxide on α -monochlorohydrin in cold methyl-alcoholic solution, forms a colourless, pleasant ethereal-smelling liquid, less viscid than glycerol. It has b. p. $196^\circ/728$ mm., $115-120^\circ/18$ mm.

Dimethylcobaltosulphate, $\text{CoSO}_4\cdot 2\text{CH}_3\cdot\text{O}\cdot\text{CH}_2\cdot\text{C}_2\text{H}_5(\text{OH})_2$, prepared by mixing the components in the cold and precipitating the saturated solution with ether, is obtained as a dark red, microcrystalline mass, which is very hygroscopic.

Glycerol gives rise to compounds with metallic sulphates having the general formula $\left[\text{M} \left(\begin{smallmatrix} \text{HO}\cdot\text{CH}_2 \\ \text{HO}\cdot\text{CH}\cdot\text{CH}_2\cdot\text{OH} \end{smallmatrix} \right)_3 \right] \text{SO}_4\cdot\text{H}_2\text{O}$.

They are obtained by heating glycerol on the water-bath with the hydrated salt, and precipitated from the saturated solution by means of alcohol.

Triglycerolcuprisulphate is a clear, blue mass of the consistence of colophony. *Triglycerolcobaltosulphate* forms a red, gelatinous mass, which melts on heating; it dissolves in water, and is reprecipitated unchanged by alcohol. *Triglycerolnickelosulphate* is obtained as a transparent, emerald-green, glass-like mass, which is very hygroscopic, but extremely stable. Barium chloride removes the sulphate from this compound, and the corresponding *chloride* results, which is very similar in appearance. *Triglycerolzincsulphate* is an amorphous, colourless mass.

Triglycerol calcium hydroxide forms slightly yellow, granular crystals, which are very hygroscopic and absorb carbon dioxide with great avidity. It dissolves easily in water, sparingly in methyl or ethyl alcohol, all three solutions having an alkaline reaction.

The compounds formed between borates and glycerol in the ordinary analytical procedure are considered to be complex salts of the above type, and not salts of the hypothetical glyceroboric acid.

On mixing boric acid, glycerol and lithium carbonate, and precipitating with alcoholic silver nitrate, bright yellow crystals of the *niter* salt, $\text{AgBO}_2\cdot 2\text{C}_2\text{H}_5(\text{OH})_3$, are obtained.

$\alpha\beta$ Glycols, when treated with hydrogen iodide, are only partly converted into alkyl iodide, the rest being reduced to hydrocarbon. They cannot therefore be estimated in this manner.

E. F. A.

Alkylated Halohydrin and Vinylethyl Ethers. JOSEF HOUBEN (*Ber.*, 1908, 41, 3708—3712).—Polemical. A reply to Hoering (this vol., i, 497).

J. J. S.

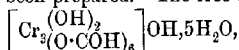
Metallic Salts of Organic Acids. I. Formates and Acetates of Chromium. ALFRED WERNER [with J. JOVANOVIĆ, S. ASCHKINASY, and J. POSSELT] (*Ber.*, 1908, 41, 3447—3465).—The

author has commenced a systematic examination of the capacity of the metallic salts of organic acids to form complex compounds. The present communication deals mainly with the formates and acetates of chromium. Two types of salts have been obtained: (a) normal salts; (b) complex salts.

The normal salts are prepared by triturating *dihydroxotetra-aquochromium sulphate*, $\left[\text{Cr} \begin{smallmatrix} (\text{OH})_2 \\ (\text{OH}_2)_4 \end{smallmatrix} \right]_2 \text{SO}_4$ (which is obtained by the addition of pyridine to an aqueous solution of chromium alum and sodium sulphate), with an organic acid. In this way *hexa-aquochromium formate*, *acetate*, *propionate*, and *butyrate* have been prepared, $[\text{Cr}(\text{H}_2\text{O})_6](\text{O}\cdot\text{CO}\cdot\text{R})_3$. The colour of these salts varies in a remarkable way, the formate being greyish-green, the acetate bluish-violet, the propionate Turkey-blue, and the butyrate silver-grey. The acetate is identical with Recoura's compound (Abstr., 1899, ii, 661), but contains 6 instead of 5 H_2O . All the salts are hydrolytically dissociated by excess of water, giving bluish-green solutions, from which sodium sulphate precipitates dihydroxotetra-aquochromium sulphate. By trituration with mineral acids, the salts are converted into the corresponding hexa-aquochromium salt of the mineral acid, proving that the organic acid group is in the ionogenic state.

The complex salts are of the type $\left[\text{Cr}_3 \begin{smallmatrix} (\text{OH})_2 \\ (\text{O}\cdot\text{CO}\cdot\text{R})_6 \end{smallmatrix} \right] \text{X}_x \cdot z\text{H}_2\text{O}$, the constitution of the metallic complex being as yet unknown.

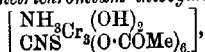
Hexaformatodioltrichromium formate, $\left[\text{Cr}_3 \begin{smallmatrix} (\text{OH})_2 \\ (\text{O}\cdot\text{COH})_6 \end{smallmatrix} \right] \text{O}\cdot\text{COH}\cdot 5\text{H}_2\text{O}$, is prepared by dissolving freshly-precipitated, moist chromium hydroxide in formic acid, D 1.15—1.20 (compare Häussermann, Abstr., 1895, i, 15). By solution in concentrated nitric acid, the air-dried formate is converted into the *nitrate*, $\left[\text{Cr}_3 \begin{smallmatrix} (\text{OH})_2 \\ (\text{O}\cdot\text{COH})_6 \end{smallmatrix} \right] \text{NO}_3 \cdot 5\text{H}_2\text{O}$, from which the corresponding *chloride*, *bromide*, *iodide*, *sulphate*, *dithionate*, and *aurichloride* have been prepared. The free base,



obtained from the nitrate and ammonium hydroxide, crystallises in light green needles.

Hexa-acetatodioltrichromium nitrate, $\left[\text{Cr}_3 \begin{smallmatrix} (\text{OH})_2 \\ (\text{O}\cdot\text{COMe})_6 \end{smallmatrix} \right] \text{NO}_3 \cdot 6\text{H}_2\text{O}$, is prepared by dissolving equal quantities of recently-precipitated chromium hydroxide in nitric acid (1 mol.) and in glacial acetic acid (6 mols.) respectively, and heating the mixed solutions on the water-bath until a pure dark green colour is produced; the nitrate slowly separates, after cooling, in tufts of dark green leaflets. The corresponding *chloride*, *bromide*, *iodide*, *periodide*, *aurichloride*, and *platinichloride* are described; the last three salts contain only $5\text{H}_2\text{O}$. *Hexa-propionatodioltrichromium chloride*, $\left[\text{Cr}_3 \begin{smallmatrix} (\text{OH})_2 \\ (\text{O}\cdot\text{CO}\cdot\text{C}_2\text{H}_5)_6 \end{smallmatrix} \right] \text{Cl} \cdot 5\text{H}_2\text{O}$, forms dark green crystals.

Hexa-acetatodiolamminetrichromium thiocyanate,

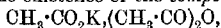


prepared from chloropentamminechromium chloride, aqueous potassium thiocyanate, and glacial acetic acid, separates from acetone in tufts of glistening, pale green needles. The thiocyanate group is probably within the metallic complex, since an aqueous solution of the salt does not immediately precipitate silver thiocyanate by treatment with silver nitrate. The mother liquor contains *hexa-acetatodiol-aquo-amminetricobalt nitrate*, $\left[\text{NH}_3\text{Cr}(\text{OH})_2 \left(\text{H}_2\text{O} \right)_2 (\text{O-COMe})_6 \right] \text{NO}_3$, which is also obtained by oxidising the thiocyanate with concentrated nitric acid; the corresponding *iodide* is also described. C. S.

Double Compounds of Acetates with Acetic Anhydride.
HARTWIG FRANZEN (*Ber.*, 1908, 41, 3641—3644).—When a recently fused and powdered acetate is boiled for a short time with acetic anhydride and the filtered solution allowed to cool, crystals of a compound of the acetate and anhydride separate. Two series of compounds appear to exist, namely, $(\text{Ac}\cdot\text{OR})_2\cdot\text{Ac}_2\text{O}$ and $\text{Ac}\cdot\text{OR}\cdot\text{Ac}_2\text{O}$; the latter have been obtained only in the case of potassium and sodium acetate. The conditions according to which the one or the other compound is produced are unknown in the case of sodium acetate, but in general the compound $(\text{Ac}\cdot\text{ONa})_2\cdot\text{Ac}_2\text{O}$ is formed when crystallisation begins in the warm solution, whilst the compound $\text{Ac}\cdot\text{ONa}\cdot\text{Ac}_2\text{O}$ is obtained when crystallisation is started in a super-cooled solution. The acetic anhydride may be combined with the acetate in a manner similar to that of water of crystallisation, or the compounds may be regarded as salts of acetylorthoacetic acids, thus: $\text{NaO}\cdot\text{CMe}(\text{OAc})_2$ and $\text{CMe}(\text{ONa})_2\cdot\text{O}\cdot\text{CMe}(\text{OAc})_2$. This explanation may throw some light on the constitution of bimolecular fatty acids; thus bimolecular acetic acid may be an acetylorthoacetic acid, $\text{CMe}(\text{OH})_2\cdot\text{OAc}$.

The *compound*, $(\text{CH}_3\cdot\text{CO}_2\text{Na})_2\cdot(\text{CH}_3\cdot\text{CO})_2\text{O}$, forms compact, colourless crystals, m. p. 160° , becoming solid on further heating. It loses acetic anhydride gradually in a vacuum desiccator. The *compound*, $\text{CH}_3\cdot\text{CO}_2\text{Na}\cdot(\text{CH}_3\cdot\text{CO})_2\text{O}$, forms slender, colourless, matted needles, sintering at 80° , m. p. 150 — 160° ; at 180 — 200° , the substance boils and begins to solidify, and the whole is completely solid at 220° . It loses acetic anhydride rapidly in a vacuum desiccator.

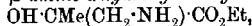
The *compound*, $(\text{CH}_3\cdot\text{CO}_2\text{K})_2\cdot(\text{CH}_3\cdot\text{CO})_2\text{O}$, forms slender, colourless crystals; evidence of the existence of the *compound*,



is also adduced. The *compound*, $(\text{CH}_3\cdot\text{CO}_2\text{Rb})_2\cdot(\text{CH}_3\cdot\text{CO})_2\text{O}$, forms slender, colourless crystals. The *compound*, $(\text{CH}_3\cdot\text{CO}_2\text{Cs})_2\cdot(\text{CH}_3\cdot\text{CO})_2\text{O}$, is also obtained in colourless crystals. J. C. C.

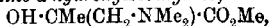
Preparation of the Esters of the Amino- α -hydroxy-acids.
LES ETABLISSEMENTS POULENC FRERES and ERNEST FOURNEAU (D.R.-P. 198306).—The esters of the amino- α -hydroxy-acids having the general formula $\text{NRR}'\cdot\text{CH}_2\cdot\text{CMe}(\text{OH})\cdot\text{CO}_2\text{R}''$ (where R'' is an alkyl group and R and R' either hydrogen atoms or alkyl groups) are produced by the esterification of the corresponding acid with aliphatic alcohols in the presence of mineral acids. The starting point in these syntheses

is chloroacetone, which, when treated successively with hydrocyanic acid and hydrolytic agents, furnishes β -chloro- α -hydroxyisobutyric acid, $\text{OH}\cdot\text{CMe}(\text{CH}_2\text{Cl})\cdot\text{CO}_2\text{H}$. This chloro-acid, on treatment under pressure with ammonia or primary or secondary aliphatic amines, gives rise to a series of amino- α -hydroxyisobutyric acids. The following compounds are described: *Ethyl β -amino- α -hydroxyisobutyrate*,

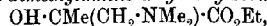


oil, b. p. $107-109^\circ/15$ mm.; *hydrochloride*, needles, m. p. 105° ; *urethane derivative*, $\text{OH}\cdot\text{CMe}(\text{CH}_2\cdot\text{NH}\cdot\text{CO}_2\text{Et})\cdot\text{CO}_2\text{Et}$, b. p. $164-165^\circ/16$ mm.

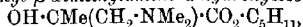
Methyl β -dimethylamino- α -hydroxyisobutyrate,



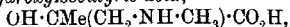
oil, b. p. $107^\circ/35$ mm.; *hydrochloride* of its *benzoyl derivative*, m. p. $149-150^\circ$. *Ethyl β -dimethylamino- α -hydroxyisobutyrate*,



oil, $88-89^\circ/16$ mm., $108-110^\circ/32$ mm.; the *hydrochlorides* of its *benzoyl* and *isovaleryl* derivatives melt respectively at 137° and $134-135^\circ$. *isoAmyl β -dimethylamino- α -hydroxyisobutyrate*,



b. p. $121^\circ/12$ mm.; the *hydrochloride* of its *benzoyl derivative*, m. p. 134° . *β -Methylamino- α -hydroxyisobutyric acid*,



m. p. $230-231^\circ$; *ethyl ester*, b. p. $112^\circ/38$ mm.

β -Diethylamino- α -hydroxyisobutyric acid, $\text{OH}\cdot\text{CMe}(\text{CH}_2\cdot\text{NEt}_2)\cdot\text{CO}_2\text{H}$, transparent prisms, m. p. 223° ; *ethyl ester*, m. p. $212^\circ/760$ mm.

Propyl β -dimethylamino- α -hydroxyisobutyrate, b. p. $94^\circ/13$ mm.; *hydrobromide* of its *isovaleryl derivative*, m. p. 165° . G. T. M.

Oxalyl Chloride. HERMANN STAUDINGER (*Ber.*, 1908, 41, 3558-3566).—Oxalyl chloride (Fauconnier, *Abstr.*, 1892, 588) is obtained in a 50% yield by the action of phosphorus pentachloride (2 mols.) on anhydrous oxalic acid (1 mol.). It is considered that the intermediate product of the reaction cannot be the half-chloride, as this is unstable at -80° , but may be a compound, $\text{C}_2\text{O}_3(\text{OPCl}_2)_2$, corresponding with that of oxalic acid with antimony pentachloride (Rosenheim and Loewenstamm, *Abstr.*, 1902, i, 358). Oxalyl chloride forms white crystals, m. p. -12° , b. p. $64^\circ/763$ mm., reacts quantitatively with water, yielding carbon dioxide, carbon monoxide, and hydrogen chloride, but forms oxalic acid when treated in the gaseous state with water vapour, and is converted into oxalates by alcohols and into oxamides by amines. It is stable towards fuming sulphuric acid, but decomposes, forming carbonyl chloride and carbon monoxide, when distilled through a glass tube heated at 600° , or when boiled with aluminium chloride in carbon disulphide solution, and may therefore be employed in place of carbonyl chloride in the Friedel-Crafts synthesis. When treated with hydrogen sulphide in ethereal solution, oxalyl chloride remains unchanged, but if quinoline is added, carbon monoxide is evolved.

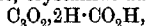
The action of ethyl mercaptan on oxalyl chloride in cold ethereal solution leads to the formation of *ethyl dithio-oxalate*, $\text{C}_2\text{O}_2(\text{SEt})_2$, which is obtained as a viscid, yellow oil, b. p. $238-240^\circ/757$ mm., solidifying at $24-25^\circ$ to light yellow crystals. G. Y.

Electrolytic Decomposition of Dicarboxylic Organic Acids:
Pimelic Acid. B. LINO VANZETTI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 331—333. Compare Abstr., 1907, i, 823).—When neutral salts of the normal acids of the oxalic acid series are electrolysed, lengthening of the carbon atom-chain of the acids is accompanied by increasing complexity of the secondary phenomena of anodic oxidation. Synthesis occurs, owing to the demolition of the anion as it becomes discharged at the anode, alcohols and unsaturated acids being formed, together with their esterification products, hydroxy-acids and lactones, and substances containing aldehydic and ketonic carbonyl groups, formed by the action of the anodic oxygen. Further, with succinic acid, ethylene is formed in appreciable quantity, whilst glutaric acid yields a little propylene, and adipic acid small amounts of two butylenes having normal carbon atom-chains; suberic acid, however, does not yield hydrocarbons in appreciable quantity. Variation of the conditions of electrolysis never leads to the formation of the corresponding cyclic hydrocarbons, tri-, tetra-, and hexa-methylene, which would be expected if the two CO_2 groups of the anion leave the latter simultaneously.

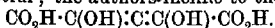
In the case of pimelic acid, now studied, the electrolytic decomposition products consist of ethylene and propylene in approximately equal proportions, and of other compounds analogous with those formed with suberic acid. No *cyclopentane* is obtained, although the closing of the carbon atom-ring should take place more readily with five carbon atoms than in the other cases. T. H. P.

Diethylmalonic Anhydride. HERMANN STAUDINGER and E. OTT (*Ber.*, 1908, 41, 3829—3830).—By purifying crude diethylmalonic anhydride by extraction with ether during forty-eight hours, it has decomp. 95—97°, and gives only a very slight depression of the freezing point in benzene solution. It is therefore very highly polymerised, and the conclusion previously drawn that it is higher than duodecimolecular is confirmed (this vol., i, 603). W. R. *

Carbon Suboxide. IV. OTTO DIELS and LEONID LALIN (*Ber.*, 1908, 41, 3426—3434).—Carbon suboxide shows little tendency to combine with aldehydes, hydrogen cyanide, or the Schiff bases; additive compounds with formic acid and with acetic acid have been obtained. In liquid sulphur dioxide at -40° to -50° , carbon suboxide and formic acid combine to give a white, crystalline *additive* compound,



which is stable in the absence of moisture, and can be recrystallised from acetyl chloride, formic acid, or methylal and light petroleum. Water instantly decomposes the substance, formic and malonic acids being formed. The molecular formula is established by analysis and by titration with sodium hydroxide (4 equivalents). The constitutional formula is still doubtful; the authors incline to the formula



The substance melts at 78° , gives off a gas, mainly carbon monoxide, just above the m. p., resolidifies, and again melts at 133 — 134° , the

m. p. of malonic acid; various other products, however, are also obtained.

The additive compound of carbon suboxide and acetic acid, obtained in a similar manner, has not yet been thoroughly examined. It is a faintly coloured syrup, which, in a vacuum at the ordinary temperature, evolves acetic anhydride, leaving a viscous residue, probably malonic anhydride, $\text{CH}_2 \begin{smallmatrix} \text{CO} \\ \diagup \quad \diagdown \\ \text{CO} \end{smallmatrix} \text{O}$, since it is converted by water into nearly the quantitative amount of malonic acid.

C. S.

Preparation of Arylthioglycolic [Arylthiolacetic] Acids. KALLE & Co. (D.R.-P. 199619).—Aromatic compounds containing a reactive halogen or nitro-group interact with alkali thiolaacetates to give rise to arylthiolacetic acids, in which the reactive substituent is displaced by the radicle $\text{S} \cdot \text{CH}_2 \cdot \text{CO}_2\text{H}$.

p-Nitrophenylthiolacetic acid, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{S} \cdot \text{CH}_2 \cdot \text{CO}_2\text{H}$, yellow needles, m. p. 152° , was obtained by heating in alcoholic soda solution, *p*-chloronitrobenzene and thiolacetic acid.

o-Nitrophenylthiolacetic acid, m. p. 157° , was similarly prepared from *o*-chloronitrobenzene.

2:4-Dinitrophenylthiolacetic acid, yellow needles, m. p. $167-168^\circ$, was produced from 1-chloro-2:4-dinitrobenzene. The crude dinitrobenzene from the mother liquors of crystallised *m*-dinitrobenzene, when condensed in this way with α -thiolacetic acid, gave rise to *o*-nitrophenylthiolacetic acid.

G. T. M.

New Reaction of Formaldehyde. F. TODTENHAUPT (*Chem. Zeit.*, 1908, 32, 1045).—When a strong solution of formaldehyde is added to a concentrated ammonium sulphide solution, the mixture becomes heated, and, after a few seconds, there separates a white, soft, sticky substance which, when cooled, forms white, glassy, resinous material with a lustrous fracture; m. p. about 45° . It is soluble in hot glacial acetic acid and in chloroform, and from the former solution, long, white needles separate, m. p. 208° . When heated, the substance (m. p. 45°) has the property of dissolving large quantities of sulphur, becoming brown in colour.

J. V. E.

Hyposulphites. V. Rongalitic Acid, Bases, and Formaldehyde. ARTHUR BINZ and ED. ISAAC (*Ber.*, 1908, 41, 3381—3387. Compare Reinking, Dehnelt, and Labhardt, *Abstr.*, 1905, i, 261).—Sodium formaldehydesulphoxylate is termed *Rongalite* by the Badische Anilin- & Soda-Fabrik, and the author uses the name *Rongalitic acid* for the corresponding acid.

When aqueous solutions of ammonium chloride, rongalite, and formaldehyde are mixed, a white, powdery substance, $\text{C}_3\text{H}_5\text{O}_3\text{N}_2\text{S}_2$, probably $\text{CH}_2 \begin{smallmatrix} \text{N}(\text{CH}_2 \cdot \text{OH}) \cdot \text{CH}_2 \cdot \text{O} \cdot \text{S} \\ \diagup \quad \diagdown \\ \text{N}(\text{CH}_2 \cdot \text{OH}) \cdot \text{CH}_2 \cdot \text{O} \cdot \text{S} \end{smallmatrix} \text{O}$, is formed.

It decomposes between 135° and 140° , reduces indigo-carmin, and when kept for some time undergoes spontaneous decomposition, yielding sulphur dioxide. It is also decomposed by hot water. When suspended in boiling alcohol and decomposed by the addition of a

small amount of water, it yields aminomethylsulphurous acid, m. p. 193° (compare Reinking, Dehnelt, and Labhardt, *loc. cit.*).

Rongalite, aniline hydrochloride, and formaldehyde react in aqueous solution and yield a colourless compound, $C_{25}H_{28}O_4N_4S_2$, m. p. 138–142°, which rapidly decomposes and turns yellow. The compound appears to be formed by the action of rongalite on the hydrochloride of anhydro-*p*-aminobenzyl alcohol, obtained by the action of formaldehyde on aniline hydrochloride. The salts of other primary amines behave in much the same manner as aniline hydrochloride.

Aqueous solutions of rongalite and aniline hydrochloride react in the absence of formaldehyde, and when the mixture is heated in a boiling water-bath it yields a colourless precipitate, $C_{27}H_{32}O_6N_4S_2$, m. p. 159–160°. When thoroughly dried, it remains colourless, but otherwise turns yellow. It does not reduce indigo-carmin, and when heated with acetic anhydride and water yields diacetaminodiphenylmethane.

Dimethylaniline hydrochloride, rongalite, and formaldehyde, when heated together in aqueous solution, yield a base, tetramethyldiaminobenzylphenylsulphone, $C_{17}H_{20}O_2N_4S$, which crystallises from alcohol or benzene in long needles, m. p. 199°. It does not reduce indigo-carmin, but reacts with nitrous acid, yielding a nitro-nitroso-derivative, $C_{16}H_{18}O_4N_4S$,

m. p. 170°.

J. J. S.

Action of Bromine on Ether. Bromoacetaldehyde. CHARLES MAUGUIN (*Compt. rend.*, 1908, 147, 747–749).—The action of dry bromine on dry ethyl ether has been investigated by Löwig (*Ann. Phys. Chem.*, 1829, 16, 376), Völckel (*Annalen* 1842, 41, 119), and Schützenberger (*Bull. Soc. chim.*, 1873, [ii], 19, 8). The author has studied the reaction in the presence of water. A mixture of ethyl ether (60 grams), bromine (10 grams), and water (30 grams) is decolorised by one hour's exposure to July sunlight. The velocity of the reaction depends on the intensity of the light (it does not occur in the dark), and is diminished by reducing the quantity of water present. When the reaction is complete, the ethereal layer gives the reactions of an aldehyde, and reacts with urethane, forming bromoacetaldehyde urethane, m. p. 146°, $CH_2Br \cdot CH \cdot (NH \cdot CO_2Et)_2$, described by Biscoff (*Ber.*, 1872, 5, 85) and by Hantzsch (*Abstr.*, 1894, i, 363). This urethane gives the aldehyde on distillation with 10% sulphuric acid, and the latter is identified by transformation into glycollaldehyde and glyoxalosazone in the manner described by Fischer and Landsteiner (*Abstr.*, 1892, 1424). The reaction affords a method of readily preparing bromoacetaldehyde.

E. H.

The Tricarbon Series. ALFRED WOHL (*Ber.*, 1908, 41, 3599–3612).—Studies in the synthesis of oxygenated acyclic derivatives of propane.

1. *Derivatives of Propaldehyde.*—[With H. SCHWEITZER].—6-Hydroxypropaldehyde, obtained in small yield by hydrolysis of 6-hydroxypropaldehydediethylacetal (*Abstr.*, 1900, i, 627) by means of dilute sulphuric acid, has b. p. 75–78°/12 mm. (corr.) (90°/10 mm. :

Nef, Abstr., 1905, i, 3), reduces cold ammoniacal silver nitrate solutions, but not Fehling's solution until after the lapse of some days, when it has commenced to undergo condensation with formation of $\alpha\beta$ -unsaturated aldehydes.

β -Iodopropaldehydedimethylacetal, $\text{CH}_3\text{I}\cdot\text{CH}_2\cdot\text{CH}(\text{OMe})_2$, prepared by heating the β -chloro-compound with potassium iodide in methyl-alcoholic solution in a sealed tube at 100° , forms a colourless oil, b. p. $85^\circ/60$ mm., has an intense odour, and is comparatively stable.

α -Bromopropaldehydedimethylacetal, $\text{CHMeBr}\cdot\text{CH}(\text{OMe})_2$, prepared by bromination of propaldehydedimethylacetal, is obtained as an oil, b. p. $52^\circ/12$ mm. Attempts to prepare α -hydroxypropaldehydeacetal from this or from the diethylacetal were unsuccessful.

2. *Derivatives of $\alpha\alpha$ -Dichloroacetone.*—[With A. KÖPPEN.]— $\alpha\alpha$ -Dichloroacetonedithylacetal, $\text{CHCl}_2\cdot\text{C}(\text{OEt}_2)\cdot\text{CH}_3$, prepared by Claisen's method (Abstr., 1898, i, 421), is obtained as a liquid, b. p. $76^\circ/8$ mm. or 183 — $184^\circ/767$ mm., has an odour resembling camphor, and is miscible with the usual organic solvents.

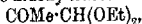
$\alpha\alpha$ -Dichloroacetonedimethylacetal, $\text{C}_5\text{H}_{10}\text{O}_2\text{Cl}_2$, forms a colourless liquid, b. p. $63^\circ/9$ mm. or 170 — $171^\circ/767$ mm., and resembles the diethylacetal.

3. *Derivatives of Dichloroisopropyl Alcohol.*—[With H. ROTH.]—The action of sodium methoxide or ethoxide on dichloroisopropyl alcohol leads to the formation of mixtures of derivatives containing varying amounts of chlorine. The benzoyl derivative, formed by heating the alcohol with benzoyl chloride, has b. p. 92 — $94^\circ/0.15$ — 0.17 mm.; this also, when treated with sodium methoxide, yields mixtures of products which cannot be separated.

s -Tetrachlorodisopropylformal, $\text{CH}_2(\text{O}\cdot\text{CHMe}\cdot\text{CHCl}_2)_2$, prepared by Henry's method (Abstr., 1905, i, 558), has b. p. $81^\circ/0.2$ mm., and when treated with sodium methoxide or ethoxide yields a product, b. p. 80 — 300° (indefinite), which reduces Fehling's solution and decolorises permanganate.

4. *Lactaldehyde.*—[With M. LANGE.]—Lactaldehyde, $\text{C}_3\text{H}_4\text{O}_2$, obtained by hydrolysis of lactaldehydeacetal (see following abstract) by means of dilute sulphuric acid, crystallises in flat needles, sinters at 101° , m. p. 105° on prolonged heating, has a bitter flavour, gives a yellow to brown coloration with alkalis, and reduces Fehling's and Tollen's solutions in the cold. When heated under 9 mm. pressure in a bath at 115 — 125° , the aldehyde distils, chiefly at 58 — 66° , yielding a mobile distillate which rapidly becomes viscid, undergoing polymerisation. Rosaniline gives a red coloration with the freshly distilled aldehyde in ten seconds, with the crystallised aldehyde in one minute, and with the viscid polymeride, seven days after distillation, in two minutes; the three colorations are of equal depth in ten minutes. In freshly prepared aqueous solution, the aldehyde is bimolecular, and gradually becomes unimolecular, slowly at the ordinary temperature, but more quickly when heated. The phenylhydrazone, prepared from the acetal, crystallises in prismatic leaflets, m. p. 90 — 92° , or when prepared from the aldehyde, m. p. 128 — 129° ; the corresponding acetol, m. p. 97 — 98° . The osazone is identical with methylglyoxalosazone. The nitrophenylhydrazone crystallises in yellow prisms, m. p. 124° (corr.); the corresponding acetol, m. p. 190 — 191° . G. Y.

Synthesis of Lactaldehyde. ALFRED WOHL and M. LANGE (*Ber.*, 1908, 41, 3612—3620).—Ethyl diethoxyacetate is obtained in a 69–70% yield from dichloroacetic acid by a modification of Schreiber's method (*Jahresb.*, 1870, 641), or in a 52% yield by the action of alcoholic hydrogen chloride on calcium glyoxylate. The *piperidide*, $\text{CH}(\text{OEt})_2 \cdot \text{CO} \cdot \text{C}_6\text{H}_{10}$, prepared by boiling the ester with piperidine, forms a viscid, colourless, strongly refracting oil, b. p. 87–90°/0.12–0.15 mm., and when treated with magnesium methyl iodide in ethereal solution yields the aldehydeacetal of methylglyoxal,



which is obtained as a colourless liquid, b. p. 54–55°/13–15 mm. (corr.) or 161.7–161.8°/761 mm. (corr.), D 0.937. In sodium hydrogen carbonate solution, it forms a *monosemicarbazone*, white needles, m. p. 132°, and in acid solution, a *disemicarbazone*, m. p. 254° (corr.) (compare Harries and Türk, *Abstr.*, 1905, i, 413). With phenylhydrazine in hot acetic acid solution, the acetal yields methylglyoxal-*osazone*. When treated with alkalis, the acetal forms a *polymeride*, b. p. 158–159°/16 mm., and on reduction with sodium and alcohol yields *lactaldehydeacetal*, which is obtained as a clear, mobile liquid, b. p. 67°/12–13 mm. (corr.) or 167–170°/758 mm. (corr.) (slight decomp.). G. Y.

Desmotropy and Merotropy. ARTHUR MICHAEL (*Annalen*, 1908, 363, 20–35).—An attempt to refer to the laws of entropy, the phenomena of desmotropy, the existence of two isomerides which can change each into the other by intramolecular wandering of a hydrogen atom, and of merotropy, the formation of derivatives of an isomeride by a substance of stable constitution. It is considered that a correct theoretical view of the whole field can be gained only if the two classes of phenomena are kept strictly separated (compare following abstracts). G. Y.

Tertiary Amines as Reagents for Distinguishing between Stable Enolic and Ketonic Derivatives. ARTHUR MICHAEL and HOWARD D. SMITH (*Annalen*, 1908, 363, 36–63).—Hantzsch and Dollfus drew attention (*Abstr.*, 1902, i, 223, 675) to the advantage of employing trimethylamine instead of ammonia as a reagent for the recognition of ψ -acids, as an addition of the base to a carbonyl group appeared improbable. They found, however, that their expectations as to the solubilities of trimethylammonium salts in ether and benzene were in many cases not fulfilled. In the present work a rise in temperature on addition of the substance to the reagent, and the formation of a new compound, are taken as the signs of chemical reaction. This method is independent of the solubilities of the substances concerned, and permits of the study of the problem without the introduction of complications by the use of a solvent. As triethylphosphine and trimethylamine unite with carbon disulphide, the formation of similar additive compounds of tertiary amines with aldehydes and ketones was not impossible, but is found not to take place.

A number of stable enolic and ketonic compounds have been investigated as to their behaviour towards triethyl-, tripropyl-, tri-

methyl-, triisobutyl-, triisobutyl-, and tribenzyl-amines. These bases are found to be incapable of enolising stable ketonic compounds. They differ in their capacity to form ammonium salts with enolic compounds, as this property depends on their structure. This capacity is found to diminish in the order in which the bases are named: triisobutylamine reacts only with the most acid enolic compounds, whilst tribenzylamine does not in any case form an ammonium salt. Triethyl- and tripropyl-amines are to be preferred as reagents, because the formation of their additive compounds is accompanied by the development of the greatest amounts of heat, and because they form the most stable salts, an advantage in the case of feebly acid enolic compounds.

The behaviour of merotropic compounds towards amines is discussed, and the conclusion is drawn that a merotropic compound has the enolic form if it reacts with a tertiary amine in absence of a solvent, and the original substance is again formed by the action of hydrochloric acid on the product, but that it has the ketonic form if, whilst not reacting with tertiary amines, it yields with primary and secondary amines, products from which the parent substance is again formed by treatment with hydrochloric acid.

The matter is less simple in the case of desmotropic compounds. The experimental results show that, as was to be expected on theoretical grounds when the conditions of stability of the enolic and the ketonic forms approximate to each other ($K \rightleftharpoons E$), the ketonic form is enolised by tertiary amines, and consequently forms ammonium salts. The action of triethylamine on the ketonic form of dibenzoyl-acetylmethane leads to the formation of an unstable ammonium salt of the enolic form, which undergoes intramolecular transformation into a stable salt, also of the enolic form. It is considered that the tertiary base acts in the first place on the acetyl carbonyl, which possesses the most energy (see Claisen, *Abstr.*, 1896, i, 557), forming the salt, $\text{CBz}_2\text{:CMe}\cdot\text{O}\cdot\text{NHEt}_3$, the tendency towards better intramolecular neutralisation then causing the triethylammonium group to wander to the benzoyl carbonyl, $\text{CBzAc:CPh}\cdot\text{O}\cdot\text{NHEt}_3$. As was to be expected, the enolic form of dibenzoylacetylmethane forms only one salt with triethylamine.

The behaviour of desmotropic compounds of the group $K \rightleftharpoons E$ towards tertiary amines agrees with the theoretical expectation, the enolic forms being converted into the ketonic. The ketonic forms of ethyl diacetyl- and dibenzoyl-succinates contain each two asymmetric carbon atoms; Knorr considered (*Abstr.*, 1899, i, 673) therefore that the two forms in which each of these esters are obtained are racemic and inactive modifications. This view is tenable no longer, as Michael and Murphy found (*Abstr.*, 1906, i, 179) that the ketonic modification of dibenzoylacetylmethane, which does not contain an asymmetric carbon atom, also exists in two forms. The ketonic form of ethyl diacetylsuccinate behaves in the same manner as that of dibenzoylacetylmethane towards tertiary amines; the modification melting at the higher temperature is converted, completely by triethylamine, partly by triisobutylamine, into the modification melting at the lower temperature. The enolic modification of ethyl dibenzoyl-

succinate, which is only slowly and partly ketonised by sodium carbonate solution (Knorr, *loc. cit.*), is transformed rapidly by traces of triethylamine, forming a mixture of the two ketonic forms. The conditions of stability of the ketones are the converse of those of the acetyl compounds, in that the modification, m. p. 75°, is transformed by triethylamine partly into the modification, m. p. 128°, whereas this is stable towards triethylamine.

The question whether merotropic ketones, which are stable towards tertiary amines alone, can be enolised by these in conjunction with an organic solvent has also been studied. It is found that an organic solvent has no influence on the enolisation of stable merotropic ketones at the ordinary temperature. This conclusion and the classification of many of the substances investigated as ketonic compounds are in disagreement with views which have been founded on the results of physico-chemical investigations. In the present state of their development, however, physical methods cannot be relied on for the decision of such problems, and views founded on the results obtained by these methods can be accepted as correct only when in agreement with the results of chemical investigations.

Ketones.—Diacetylmethane, diacetylene, acetylheptylmethane, benzoylacetylmethane, and dibenzoylmethane, oxalyldiacetone, oxalyldimesityl oxide, oxalyldiacetophenone, ethyl acetoacetate, methylacetoacetate, ethylacetoacetate, benzoylacetoacetate, α -diphenyl acetoacetate, benzoylsuccinate, succinylsuccinate, acetonedicarboxylate, acetylmalonanilate, and cyanoacetate are stable towards tertiary amines in the absence of a solvent. In some cases, solution takes place on heating with triethylamine or on treatment with a concentrated solution of the base, but the unchanged ketone is then regained on cooling or on evaporation at the ordinary temperature. Ethyl diacetoacetate, which is stable towards tertiary amines, reacts with primary and secondary amines, forming carbonyl *additive* compounds.

Ethyl oxalacetate is converted by tertiary amines into ethyl oxalocitrolactone (Wislicenus and Beckh, *Abstr.*, 1897, i, 397). *Methyl oxalocitrolactone*, $C_{11}H_{12}O_6$, obtained in the same manner from methyl oxalacetate, forms white crystals, m. p. 63°; the *triethylammonium*, m. p. 103°, *tripropylammonium*, m. p. 88–89°, and *barium*, m. p. 225–227°, salts are described.

Ethyl methyloxalacetate and triethylamine form, with development of heat, a brown *oil*, from which the original ester cannot be regained.

Enolic Compounds.—The following compounds react with tertiary amines with development of heat:

Ethyl α -hydroxymethylenepropionate forms unstable *salts*, which decompose in a vacuum, yielding the ester.

Hydroxymethyleneacetophenone forms oily *products*, which, on treatment with hydrochloric acid, yield a nitrogen-free *substance*, m. p. 73–74°, gradually becoming resinous.

Hydroxymethylenecamphor reacts with triethyl-, tripropyl-, and triisamyl-amines, but not with triisobutylamine, forming *oils*.

With triethylamine, ethyl phenyloxalacetate forms a colourless,

viscid oil, $(C_{14}H_{16}O_5)_2 \cdot NEt_3$, from which hydrochloric acid liberates the original ester.

Ethyl acetylpyruvate reacts with trimethylamine in benzene-toluene solution, forming a yellow oil, which solidifies when rubbed. Similar yellow oils are formed also with other tertiary amines, but only slowly with triisobutylamine. The action of hydrochloric acid on these oily salts leads to the formation of Claisen and Stylos's decomposition product (Abstr., 1887, 918), but of copper acetate in concentrated aqueous-alcoholic solution to the formation of the copper derivative of the pyruvate.

Tetric acid yields a white, crystalline triethylammonium, $(C_2H_5O_3)_2 \cdot NEt_3$, m. p. 30—35°, and an oily triisoamylammonium salt.

With tertiary bases, ethyl acetylmalonate and benzoylacetacetate form salts, which decompose slowly in a vacuum.

Ethyl dibenzoylacetate does not react with tripropyl-, triisoamyl-, or triisobutylamine, but yields a yellow, crystalline trimethylammonium, m. p. 75—80°, and a triethylammonium, $C_{18}H_{16}O_4 \cdot NEt_3$, m. p. 69—70°, salt, decomposing slowly in a vacuum.

In contrast to ethyl diacetylacetate, which is a stable lactone, diacetylacetanilide behaves as an enolic compound, forming an oily salt with triethylamine.

Triacetyl methane forms salts with trimethyl- and triethylamine, but not with other tertiary bases.

The yellow trimethylammonium salt of benzoyldiacetyl methane is unstable; the triethylammonium salt, $C_{12}H_{12}O_3 \cdot NEt_3$, m. p. 55—58°, decomposes slowly in a vacuum.

Ethyl hydroxymethyleneacetacetate and diacetylhydroxymethylene methane form moderately stable, red and dark green oily salts with triethylamine.

Methyl oxalysuccinate combines with tertiary fatty amines, with the exception of triisobutylamine, forming oils, from which hydrochloric acid liberates apparently the parent ester.

Ethyl acetonedioxyolate forms with trimethylamine a yellow salt, m. p. 81—83°, with triethyl-, tripropyl-, and triisoamylamines, yellow oils, which are decomposed by hydrochloric acid with formation of the parent ester, and with triisobutylamine in benzene, a yellow solution which deposits a red oil not decomposed by dilute hydrochloric acid.

The triethylammonium salt of ethyl acetophenoneoxalate, $C_{12}H_{12}O_4 \cdot NEt_3$, forms a white, crystalline mass, m. p. 57°. This ester combines with other tertiary amines, forming oils.

Methyl dioxalysuccinate combines with all tertiary amines, including triisobutylamine.

Phloroglucinol yields a methylammonium, m. p. 165—167°, ethylammonium, $(C_6H_6O_3)_2 \cdot NEt_3$, m. p. 103—104°, propylammonium, m. p. 87—91°, isoamylammonium, m. p. 97—100°, and triisobutylammonium, m. p. 103—107°, salt. This behaviour, in conjunction with the known reactions of phloroglucinol, is in agreement with the conception of it as a hydroxydiketotetrahydrobenzene.

Phloroglucinol diethyl ether combines with tertiary amines slowly in absence of a solvent, forming colourless oils, which are formed more rapidly by treatment of the ether with the tertiary amine in benzene and evaporation of the solution.

Ethyl phloroglucinoldicarboxylate forms a triethylammonium salt, $C_{12}H_{14}O_7 \cdot NEt_3$, m. p. 101° , which decomposes moderately quickly in a vacuum.

Enol-ketone Desmotropic Compounds.—The enolic modification of methyl mesityloxidoxalate ($K \rightleftharpoons E$) combines with triethylamine, forming a yellow, oily salt, which gradually decomposes in a vacuum, yielding the enolic ester. The ketonic modification does not react with tertiary amines.

The enolic modification of dibenzoylacetylmethane ($K \rightleftharpoons E$) reacts with trimethylamine in toluene solution at -15° , forming yellow, hexagonal prisms, m. p. $94-97^\circ$; this salt, which remains unchanged for some days at 0° in a sealed tube, but decomposes rapidly when exposed to air at the ordinary temperature, is formed also, but more slowly, by the action of the base on the ketonic modification of dibenzoylacetylmethane in toluene solution at -10° . Both modifications give a transient, yellow coloration, and, thereafter, a colourless, crystalline product when treated with the base in ethereal solution at -15° . The ketonic modification reacts with triethylamine alone or in toluene solution, forming a yellow salt, $C_{17}H_{14}O_6 \cdot NEt_3$, m. p. $86-90^\circ$; in ethereal solution, this is at first formed, but changes into colourless crystals. Tripropylamine also transforms the ketone, forming the ammonium salt of the enolic modification. On the other hand, whilst the enolic modification is indifferent to triisamyl- and triisobutylamines, these bases slowly enolise the ketone.

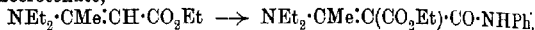
The enolic modification of tribenzoylmethane ($K \rightleftharpoons E$) forms yellow salts with trimethyl- and triethylamines; whilst the action of triisamylamine leads partly to the formation of a salt, partly to ketonisation of the enol, the latter action alone takes place in presence of triisobutylamine. The ketonic modification is indifferent to tertiary amines alone, or in presence of benzene or ether at the ordinary temperature.

The enolic modifications of ethyl benzylidenebisacetoacetate ($K \rightleftharpoons E$) are transformed by tertiary bases into the ketonic form, which is stable towards these reagents. G. Y.

Phenylcarbimide as Reagent for Determining the Constitution of Merotropic Compounds. ARTHUR MICHAEL and PHILIP H. COBB (*Annalen*, 1908, 363, 64-93).—Dieckmann, Hoppe, and Stein found (Abstr., 1905, i, 135) that the reactivity of phenylcarbimide is greatly increased by the presence of traces of an alkali, and concluded that if such traces are excluded the carbimide does not react with stable ketones, such as the acyclic 1 : 3-dicarbonyl compounds, or with hydroxymethylene derivatives, and that under these conditions phloroglucinol is not attacked, or is attacked only with great difficulty compare Goldschmidt, Abstr., 1905, i, 340). Michael, on the other hand, found phloroglucinol to react readily with phenylcarbimide in

cold ethereal solution (Abstr., 1905, i, 195). The present authors have therefore again studied the action of phenylcarbimide on ketones, carrying out the experiments partly in glass tubes boiled out with water and acids, and partly in platinum apparatus, and taking all precautions as to the purity of the substances employed. It is found that at the ordinary temperature ethyl acetoacetate and ethyl phenylhydroxymethyleneacetate form *C*- and *O*-additive compounds respectively, whilst at 100° phloroglucinol readily forms the compound obtained by Dieckmann, Hoppe, and Stein only in presence of alkalis. An observation that a specimen of phenylcarbimide containing phenylcarbylamine is less reactive than the pure reagent may explain the differences in the results obtained by the different investigators.

Only certain ketones are capable of reacting with phenylcarbimide; dibenzoylmethane, for example, is indifferent to this reagent. The chemical potential necessary for the reaction $\text{CH}_2\text{R}\cdot\text{COR}' + \text{NPh}\cdot\text{CO} = \text{NHPh}\cdot\text{CO}\cdot\text{CHR}\cdot\text{COR}'$ must depend on the nature of the substituting groups R and R'. The formation of the additive compound takes place directly, and not by way of an intermediate *O*-derivative, as is shown by the formation of an *additive* compound by ethyl β -diethylaminocrotonate,



On treatment with concentrated sulphuric acid, the additive compound yields diethylamine and the additive compound of phenylcarbimide and ethyl acetoacetate; hence addition can take place directly to an unsaturated methenyl group.

It has been found further that, whilst ethyl benzoylacetate reacts with phenylcarbimide, forming ethyl benzoylmalonanilide, ethyl methyl- and ethyl-acetoacetates are indifferent to the reagent even in presence of alkalis.

Whilst ethyl cyanoacetate does not react with phenylcarbimide, the sodiocyanoacetate readily forms an *additive* compound, which, on treatment with hydrochloric acid, yields ethyl cyanomalonanilide, $\text{C}_{12}\text{H}_{12}\text{O}_5\text{N}_2$. This crystallises in prisms, m. p. 145°, and is hydrolysed slowly by boiling alkalis, forming cyanoacetanilide.

α -Formylpropionanilide (Abstr., 1905, i, 195) separates from light petroleum in crystals, m. p. 95—96°.

A study of the rise in temperature caused by mixing fatty alcohols with phenylcarbimide gave the following results. The rise in temperature is quoted in degrees; the figures in brackets are the weights in grams of the alcohol and the phenylcarbimide brought into reaction.

Methyl alcohol (0.1 : 0.26), 37°; ethyl alcohol (0.14 : 0.29), 23°; *n*-propyl alcohol (0.1 : 0.3), 23°; (0.17 : 0.32), 28°; *isopropyl* alcohol (0.1 : 0.3), 12°; (0.17 : 0.32), 13°; *n*-butyl alcohol (0.185 : 0.3), 26.5°; (1.0 : 1.6), 47°; *isobutyl* alcohol (0.185 : 0.3), 22°; (1.0 : 1.6), 31°; *tert*-butyl alcohol (0.185 : 0.3), 5°; (1.0 : 1.6), 4°. The *additive* compounds of the three butyl alcohols crystallise from light petroleum in needles, m. p. 63—64°, 86°, and 134—135° respectively.

The action of phenylcarbimide on ethyl lactate leads to the formation of considerable amounts of carbanilide.

Methyl salicylate does not react with phenylcarbimide at 160°. At 170° the nixture yields only carbanilide, and at 175° triphenylisocyanurate. On the other hand, an *additive* compound, $\text{C}_{15}\text{H}_{15}\text{O}_4\text{N}$,

m. p. 117°, which is probably identical with Lambling's derivative Abstr., 1902, i, 756), is formed in two to four days by the action of phenylcarbimide on methyl salicylate at the ordinary temperature in presence of traces of sodium acetate, propylamine, or tributylamine. In some experiments, there was obtained a product, m. p. 238—240° (Spape, Trans., 1885, 47, 770; 1886, 49, 254; Smolka and Friedreich, Abstr., 1890, 618). Ethyl salicylate reacts in the same manner as the methyl ester. The additive compound, $C_{16}H_{15}O_4N$, formed in two hours in presence of traces of sodium acetate, separates from carbon disulphide in crystals, m. p. 98—100°.

In presence of sodium acetate, the isobutyl ester and phenylcarbimide form triphenyl isocyanurate.

In contrast to the salicylates, methyl *m*- and *p*-hydroxybenzoates react with phenylcarbimide in the absence of a catalyst at the ordinary temperature. The additive compound of the *m*-ester, $C_{16}H_{15}O_4N$, separates from benzene in crystals, m. p. 115—116°; the additive compound of the *p*-ester, m. p. 134—135°.

The authors have studied also the behaviour of phenylcarbimide towards phenols and anilines containing negative substituting groups at ortho atoms (compare Gumpert, Abstr., 1885, 656; 1886, 342). It is found that the reaction is hindered more by a nitro-group in the ortho-position than by a nitro-group in the para-position. *o*-Nitrophenol reacts with phenylcarbimide only at 150—170°, traces of an additive compound being formed. At the ordinary temperature, the carbimide slowly polymerises, and the urethane is not formed. *p*-Nitrophenol and phenylcarbimide, on the other hand, react slowly at the ordinary temperature, forming the urethane, $C_{13}H_{10}O_4N_2$, which crystallises in yellow prisms; polymerism of the reagent does not take place.

Picric acid does not react with phenylcarbimide.

Both *o*- and *p*-chlorophenols react more readily than does *p*-nitrophenol; *o*-chlorophenyl phenylcarbamate, $C_{13}H_{10}O_2NCl$, separates from benzene in crystals, m. p. 120—121°.

Quantitative experiments with the three nitroanilines show that the meta-compound reacts most easily, the ortho-compound least so. Similarly, the velocity of the formation of *o*-chlorodiphenylcarbamide, needles, m. p. 181—182°, from *o*-chloroaniline and phenylcarbimide can be followed by titration, but not that of the meta- or the para-compound, which takes place with too great rapidity.

The dihydroxybenzenes do not react with phenylcarbimide in ethereal solution at the ordinary temperature; the mixture gradually forms small amounts of the polymeride of the carbimide. G. Y.

Acetyl Chloride and Acetic Anhydride as Reagents for Distinguishing between Enolic and Ketonic Modifications. ARTHUR MICHAEL and ARTHUR MURPHY, jun. (*Annalen*, 1908, 363, 4—106).—The formation of an acetate by the action of acetyl chloride on a carbinol, $R\cdot OH$, takes place in two stages, the first being the formation of an additive compound, $R\cdot O\cdot CMeCl\cdot OH$, and the second, the splitting off of hydrogen chloride. The ease with which the first stage, and therefore the whole reaction, takes place depends on the nature of the group R , and the reaction

may be entirely prevented if R is sufficiently negative. It is for this reason that the stable enolic compounds, ethyl acetylmalonate and benzoylacetoacetate, are not acetylated by acetyl chloride or acetic anhydride, whilst benzoyldiacetylmethane remains unattacked by acetyl chloride even in pyridine solution. Similarly, the enolic forms of desmotropic compounds of the groups $K \rightleftharpoons E$ and $K \rightarrow E$ are not acetylated by acetyl chloride, but are dissolved and ketonised by the reagent. On the other hand, a ketonic modification may be acetylated by acetyl chloride if the ketone carbonyl possesses sufficient chemical potential for the chloride to form the additive compound, $\text{OAc} \cdot \text{CRCl} \cdot \text{C}_n\text{H}_{2n+1}$, and if the conditions are favourable to the splitting off of hydrogen chloride from this, when the acetyl derivative of the enolic modification must be formed; hence acetylation of a merotropic compound by acetyl chloride or acetic anhydride cannot be looked on as evidence of the existence of the substance in the enolic form.

When acetic anhydride is employed, there is the further disadvantage that reaction takes place usually only at high temperatures ($150-200^\circ$), when intramolecular transformation is still more probable, especially as it is difficult under such conditions to exclude sodium acetate, which Dieckmann, Hoppe, and Stein have shown (Abstr., 1905, i, 135) to act catalytically even in traces. This disadvantage is avoided by employing acetyl chloride at temperatures below 100° , but, although under these conditions enol-acetyl derivatives are not obtained from the ketonic modifications of di- and tri-acetylmethanes, its behaviour with certain other enolic and ketonic compounds shows that acetyl chloride cannot be considered as a trustworthy reagent for determining the constitution of a merotropic compound.

Enolic Compounds.—When heated with acetyl chloride in a reflux apparatus, ethyl acetylmalonate, benzoylacetoacetate, dibenzoylacetoacetate and *C*-acetylmalonanilide, *C*-diacetylacetanilide, benzoyldiacetylmethane, and triphenylvinol remain unchanged. Under the same conditions, ethyl α -hydroxymethylenepropionate and hydroxymethylenephénylacetate, and hydroxymethylenecamphor form acetyl derivatives. Benzoyldiacetylmethane and triphenylvinol are not acetylated by boiling acetic anhydride.

Ketonic Compounds.—Diacetylmethane, *aa*-diacetylane, acetylheptylmethane, ethyl acetoacetate and diacetoacetate, methyl oxalacetate, *C*-dibenzoylacetanilide, ethyl diphenylacetoacetate, benzoylacetylmethane, dibenzoylmethane, ethyl benzoylacetate, ethyl methane tricarboxyanilide, and ethyl benzoylsuccinate remain unchanged, or if attacked do not form acetyl derivatives when heated with acetyl chloride at 100° .

Enol-keto-desmotropism.—The enolic modification of methyl methyl oxidoxalate ($K \rightleftharpoons E$) is transformed by acetyl chloride at the ordinary temperature into the ketonic modification, which is obtained together with an oil, insoluble in sodium carbonate, when the enol is heated with acetyl chloride, traces of hydrogen chloride being evolved. Under the same conditions, the ketonic modification remains unchanged.

The ketonic modification of dibenzoylacetylmethane ($K \approx E$), m. p. 148° , is formed by the action of acetyl chloride in ethereal solution on the enolic modification, or on the ketonic modification, m. p. $107-110^\circ$.

The enolic modification of ethyl diacetylsuccinate ($K > E$) is transformed by acetyl chloride into a mixture of the β - and γ -ketonic forms; under the same conditions, the γ -ketone is transformed into the β -ketone, and hence prolonged action of acetyl chloride on the enolic modification leads to the formation of the β -ketone alone.

The ketonic modifications of tribenzoylmethane ($K \approx E$), *p*-bromotribenzoylmethane, and ethyl dibenzoylsuccinate are formed when the enolic modifications are heated with acetyl chloride. Under the same conditions, the ketonic modifications of the two methane derivatives remain unchanged, whilst the γ -ketonic form of the ester, m. p. 75° , is converted into the β -ketonic form, m. p. $128-130^\circ$.

G. Y.

Sugars. JAN. J. BLANKSMA AND W. ALBERDA VAN EKENSTEIN (*Chem. Weekblad*, 1908, 5, 777-781).—*l*-Gulose, *l*-idose, *d*-talose, and *d*-ribose have been isolated by combination with substituted phenylhydrazines, followed by decomposition of the hydrazones by benzaldehyde or formaldehyde. The sugars were obtained in the form of groups which did not crystallise.

l-Gulose was prepared by reduction of *l*-gulonolactone with sodium amalgam (compare Fischer and Stahel, Abstr., 1891, 667), the sodium hydroxide formed being neutralised periodically with sulphuric acid. After the reduction, the solution was concentrated, the sodium sulphate filtered off, and washed with alcohol to remove the syrup. Evaporation of the alcohol left a syrup containing somewhat less than half the weight of the lactone used. This substance separated out in part after a time, and the rest was removed by dissolving in water, neutralising with barium carbonate, filtering off the barium gulonate, and concentrating the filtrate. On treating the residual syrup with phenylhydrazine, *l*-gulosephenylhydrazone separated in yellow crystals, m. p. 136° . The product recrystallised from water has m. p. 143° . It was boiled in aqueous solution with the calculated quantity of benzaldehyde, the benzaldehydephenylhydrazone filtered off, and the slight excess of benzaldehyde removed by steam. On evaporation, the *l*-gulose remained as a light yellow syrup of sweet taste. It could not be obtained crystalline from either water, methyl alcohol, ethyl alcohol, mixtures of alcohol and water, or of alcohol and ether. When cooled with liquid air, it solidified to a hard, glass-like solid, m. p. -20.4° . Its power of reducing Fehling's solution is 71.5% of that of invert-sugar. These numbers are probably too low, as the syrup contained water. Fischer (*loc. cit.*) states that *l*-gulose is slightly extrorotatory; that obtained by the authors' method is levorotatory.

The methylguloside was obtained from the syrup by the action of a concentrated solution of hydrochloric acid in absolute methyl alcohol,

removal of the hydrochloric acid by lead carbonate, and evaporation of the filtrate. It is a syrup, and has not been crystallised.

Benzylguloside was prepared by a similar method from benzyl alcohol. After long standing, it crystallises, and on recrystallisation from methyl alcohol has m. p. 145° . The quantity was too small to admit of analysis.

l-Idose was obtained by a method analogous to that employed for *l*-gulose, starting from *l*-idonolactone. It is a clear syrup, with $[\alpha]_D + 7.5^{\circ}$, and a reducing power 43% of that of invert-sugar. It does not combine with substituted phenylhydrazine or benzoylhydrazine.

d-Talose was prepared from talonic acid, obtained by Fischer's method (Abstr., 1892, 299), which yields a mixture of unchanged galactonic acid and talonic acid. The two acids were converted into the corresponding phenylhydrazides, and these separated by repeated crystallisation from alcohol, talonylphenylhydrazide being the most soluble, and separating from the mother liquor in star-shaped or nodular, acicular masses. Talonic acid was obtained from its phenylhydrazide by boiling with baryta water, removing the phenylhydrazine with ether, and adding sulphuric acid (compare Fischer and Passmore, Abstr., 1890, 152). The acid was converted into its lactone by heating on the water-bath, and reduced to *d*-talose with sodium amalgam. The phenylmethylhydrazone was then prepared, and, after repeated crystallisation from methyl alcohol, had m. p. 154° . When boiled in aqueous solution with benzaldehyde, the hydrazone is converted into talose, which forms a colourless syrup, $[\alpha]_D + 13.95^{\circ}$; reducing power is 71.4% of that of invert-sugar.

l-Ribose was prepared similarly from ribonolactone, but could not be purified by conversion into its phenylhydrazone (compare Fischer and Piloty, Abstr., 1892, 439) or phenylmethylhydrazone, as these compounds are freely soluble in all solvents. The syrup has $[\alpha]_D + 14^{\circ}$, and a reducing power 55.5% of that of invert-sugar. A. J. W.

Presence of *i*-Dimethylinosite in the Latex of *Melaboei* from Sumatra. ANNE W. K. DE JONG (*Rec. trav. chim.*, 1908, 27, 257—259).—The liquid expressed from the coagulated latex of *Melaboei* contains *i*-dimethylinosite, which can be isolated in the form of rectangular crystals, m. p. 206° (Girard, *Compt. rend.*, 1874, 67, 820, gives 195°); the tetra-acetyl derivative has m. p. 195° (Girard, *loc. cit.*, gives 193°). On treatment with hydriodic acid, it yields *i*-inosite, m. p. 222° (Maquenne, Abstr., 1887, 356, gives 217°), which forms a hexa-acetyl derivative, m. p. 217° (Maquenne, Abstr., 1887, 908, gives 212°). M. A. W.

Forms of Lactose. C. S. HUDSON (*J. Amer. Chem. Soc.*, 1908, 30, 1767—1783).—In an earlier paper (Abstr., 1904, i, 974), it has been stated that lactose hydrate crystallises from aqueous solutions of the sugar at the ordinary temperature, whilst at temperatures above 95° the β -anhydrous form separates. The present investigation was undertaken with the object of determining the transition temperature between these forms of lactose, and as it was found that the work illustrated very fully the general conditions underlying transition

temperatures, the measurements were extended to several related phenomena.

All the results agreed quantitatively with the conclusions obtained previously, namely, that aqueous solutions of lactose contain two substances in equilibrium, and that the mutarotation of lactose results from the gradual establishment of equilibrium of the balanced equation $C_{12}H_{22}O_{11} \cdot H_2O \rightleftharpoons H_2O + C_{12}H_{22}O_{11}$ (β -modification). It is shown that the hypotheses of hydration and stereoisomerisation which have been advanced to explain the mutarotation of sugars are summed up and reconciled by the following equation, in which the equilibrium of the first balanced reaction is established instantaneously, whilst that of the second is attained gradually, giving rise to the slow change of rotation: α -form + $H_2O \rightleftharpoons$ hydrated form $\rightleftharpoons H_2O + \beta$ -form.

Determinations of the initial and final solubilities of lactose hydrate and β -anhydride between 0° and 100° show that the transition temperature between them is 93° , and this value is confirmed by vapour-pressure measurements. The specific rotatory power of the three forms of lactose has been determined at 15° and 25° , and the results show that the equilibrium in solution does not change perceptibly with the temperature in this interval. It has also been found that the equilibrium does not change with the concentration, since there is no slow change in the rotation of the solutions after dilution. Schmöger has found the rotatory power of the stable solution of lactose at 20° to be 55.3° per gram $C_{12}H_{22}O_{11}$. Accepting this value, the initial specific rotation of the hydrate is 36.0° , and that of the β -anhydride, 35.4° . The freezing points of lactose solutions, the vapour pressures of saturated solutions of lactose hydrate, and the aqueous vapour pressure of the solid hydrate in contact with the β -anhydride are also recorded. The heat of solution of the β -anhydride has been calculated from the solubility data, and found to agree with the value obtained by direct measurement (this vol., ii, 665). The heat of vaporisation of water from the hydrate has been calculated from the vapour pressures of the hydrate, and also from the heats of solution and transition of the two forms, and the two values agree satisfactorily. E. G.

Colloidal Properties of Starch and the Unity of its Constitution. EUGÈNE FOULARD (*Compt. rend.*, 1908, 147, 813—816. Compare his vol. i, 503).—The author has shown previously that a pseudo-solution of starch, when filtered through collodion, is divided into two portions, one consisting of a true solution, the other containing colloidal starch. The experiments described in the present paper confirm the conclusion that starch is a single chemical compound capable of undergoing complete and reversible physical transformation into the state of perfect solution. Thus, when the pseudo-solution is fractionally filtered through a collodion membrane, the successive fractions are found to have the same rotatory power. The residual fractions of the colloid show less tendency to coagulate than the earlier fractions, but this is found to be due to the fact that the traces of mineral phosphates, on the presence of which this phenomenon depends, are not evenly divided

between the true solution and the colloidal residue, the last fraction of which is almost neutral to methyl-orange or phenolphthalein.

The conclusion is drawn that the mode of aggregation of the elementary starch molecule is variable, and depends on the reaction of the surrounding plasma, which varies under different conditions. Thus, owing to different influences, such as the age of the cell-wall modifying the osmotic permeability of the membrane, different molecular aggregates of variable resistance are produced, and this gives rise to the concentric layers observed in the natural starch grain. W. O. W.

Action of Ammonium Persulphate Solution on Cellulose.
II. The Relation of the Cellulose Peroxide formed to the other Products of the Reaction and the Mechanism of the Process of Oxidation. Reaction of Oxycellulose with Nessler's Reagent. HUGO DITZ (*J. pr. Chem.*, 1908, [ii], 78, 343—364. Compare Abstr., 1907, i, 829).—As it has been stated that hydrogen peroxide is formed by the decomposition of ammonium persulphate, the action of hydrogen peroxide on cellulose was compared with that of the persulphate. Whilst the action of the latter is accompanied by evolution of much gas, only very little gas is evolved by the action of hydrogen peroxide, and cellulose peroxide is not formed. It is shown that the cellulose peroxide formed by the persulphate method does not contain sulphuric acid. When boiled with water, the peroxide yields a small amount of a flocculent substance, which may be oxycellulose; the acid (acid-cellulose?) present in the peroxide is insoluble in water. Cellulose peroxide does not evolve ammonia when boiled with lime water, but gives with Nessler's reagent a brown coloration, becoming grey in consequence of the reduction of the mercuric salt by the small amount of oxycellulose present in the peroxide. Oxycellulose contains probably an aldehyde group, as it behaves towards Nessler's reagent in the same manner as a very dilute solution of formaldehyde; it is suggested that the reducing properties of cellulose and hydrocellulose are dependent on the presence of small amounts of oxycellulose (compare Rosenthaler, Abstr., 1906, ii, 911).

The gases evolved by the action of ammonium or potassium persulphate on cellulose are now found to contain carbon dioxide as well as "active" oxygen; the latter is evolved from ammonium persulphate, also, by the action of zinc, tin, or antimony in presence of dilute sulphuric acid; copper, on the other hand, dissolves in ammonium persulphate in presence of dilute sulphuric acid without evolution of "active" oxygen or sulphur dioxide. When 100 c.c. of a 10% solution of ammonium persulphate are heated with filter paper and 5 c.c. of dilute sulphuric acid, and the gases evolved passed into a potassium iodide-starch solution, this becomes violet after eight minutes, and after fifteen minutes requires 0.3 c.c. of *N/10* sodium thiosulphate solution for decolorisation. A slightly greater effect is produced by employing linen in place of filter paper. Under the same conditions, but in absence of cellulose, the potassium iodide-starch solution is only slightly coloured in eleven minutes, and after twenty-five minutes is decolorised by 0.05 c.c. of *N/10* sodium thiosulphate. In absence of both cellulose and sulphuric acid, the gases evolved on heating ammonium

persulphate solution do not colour potassium iodide-starch solution in twenty-five minutes.

In conclusion, the mechanism of the oxidation of cellulose is discussed, and the formation of oxycellulose is compared with that of formaldehyde when methyl alcohol is heated with a persulphate. The formation of the peroxide may take place by addition of oxygen either directly to the cellulose molecule or secondarily to oxycellulose. G. Y.

Preparation of Sulphonic Esters of Cellulose. ACTIEN-GESELLSCHAFT FÜR ANILIN FABRIKATION (D.R.-P. 200334).—Alkali-soluble cellulose, produced from bleached cotton by the action of zinc chloride and hydrochloric acid, is treated with toluene-*p*-sulphonyl chloride in the presence of 10% aqueous sodium hydroxide. The toluene-*p*-sulphonic ester of cellulose separates after twenty hours as a white, amorphous powder, insoluble in acid zinc chloride solution or in cuprammonium solution; it dissolves in hot glacial acetic acid, and its solution in epichlorohydrin, chloroform, or ethyl acetate on evaporation leaves a transparent pellicle. G. T. M.

Preparation of Alcohol from Substances containing Cellulose. THEO KOERNER (*Zeitsch. angew. Chem.*, 1908, 21, 2353–2359).—The author has endeavoured to ascertain whether the sugar obtained by hydrolysis from wood, etc., by dilute sulphuric acid was entirely derived from the cellulose, or in part from the lignous substances. Comparable experiments with sawdust, straw, and sulphite-cellulose show the amount of alcohol obtained to be proportional to the cellulose present.

It has been stated that sulphurous acid increases the yield of alcohol, but the author finds that such previous treatment actually decreases the amount of alcohol obtainable. As hydrocellulose is found to yield as much as 17.95% of alcohol, it was thought possible that by treating wood, sulphite-cellulose, or straw with oxidising agents before hydrolysis, a similarly increased yield of alcohol might be obtained. With dilute solutions of hydrogen peroxide, this has been realised, the yield of alcohol being materially increased, although in the case of chromic acid a marked decrease is observed. Similarly, the yield of alcohol is found to be decreased by a preliminary oxidation with a solution of potassium persulphate or ozone, the oxidation proceeding beyond the hydrocellulose stage. J. V. E.

Investigations on the Charring of Wood. II. PETER KLASON, GUST. VON HEIDENSTAM, and EVERT NORLIN (*Arkiv. Kem. Min. Geol.*, 1908, 3, No. 10, 1–17. Compare this vol., i, 717).—The charring of wood out of contact with air at a maximum temperature of 400° proceeds mainly according to the equation $2C_{14}H_{20}O_{28} = 3C_{16}H_{10}O_2$ (wood-charcoal) + $28H_2O + 5CO_2 + 3CO + C_{28}H_{32}O_9$. The velocity of this change depends on the temperature. Charring begins at about 270°, and increases greatly in rapidity at about 300°. This dry distillation of wood is an exothermic change, the heat of the reaction being about 6% of the heat of combustion; in the case of cellulose, the heat of the reaction is about 5% of that of combustion. The methyl alcohol obtained is formed entirely from the methoxy-groups of the lignin, and the amount obtained from birch or beech wood is about

double that yielded by fir or pine. Acetic acid is formed from the cellulose, and also, to a greater extent, from the lignin; birch or beech gives about double as much of the acid as fir or pine. The calorific value of the combustible gases formed during dry distillation is about 3.8% of that of the wood itself; these gases contain neither hydrogen nor aromatic hydrocarbons. Wood charcoal, having the composition $C_{16}H_{10}O_2$, may be regarded as the product of the exothermic charring of wood.

T. H. P.

Modification of the Preparation of Methylamine from Bromoacetamide. MAURICE FRANÇOIS (*Compt. rend.*, 1908, 147, 680—682. Compare this vol., i, 768).—By a modification of the method of preparing methylamine from bromoacetamide described previously, the author is able to obtain a yield of 72% of the theoretical, instead of 35%. The solution of bromoacetamide is prepared by the interaction of acetamide and bromine in presence of water and calcium carbonate, thus avoiding possible loss of bromine through formation of hypobromites or bromates, as when potassium hydroxide is used. The resulting red solution is mixed with cold 30% aqueous sodium hydroxide, and placed in a narrow tube bent several times up and down, which is heated by boiling water. The products are distilled in a current of steam, ammonia is separated by yellow mercuric oxide, and the methylamine distilled off and converted into the hydrochloride.

J. C. C.

Preparation of Amino-alcohols. J. D. RIEDEL (D.R.-P. 199148).—The amino-alcohols, $OH \cdot CRR' \cdot CH_2 \cdot NR''R'''$, are obtained by the action of primary or secondary aliphatic amines on the alkylene oxides, $CRR' \cdot \begin{smallmatrix} CH_2 \\ | \\ O \end{smallmatrix}$.

β-Methylbutylene αβ-oxide, $CMeEt \cdot \begin{smallmatrix} CH_2 \\ | \\ O \end{smallmatrix}$, b. p. 80°/760 mm., prepared

from *β*-methylbutylene chlorohydrin and sodium hydroxide, gives rise to *dimethylaminodimethylethylcarbinol*, $NMe_2 \cdot CH_2 \cdot CMeEt \cdot OH$, b. p. 57°/23 mm., when treated with dimethylamine in benzene solution at 125°. The *hydrochloride* of the *benzoyl* derivative of this base, m. p. 175°, may be employed as a local anæsthetic.

isoButylene αβ-oxide, $CMe_2 \cdot \begin{smallmatrix} CH_2 \\ | \\ O \end{smallmatrix}$, b. p. 53°/760 mm., prepared from *isobutylene* chlorohydrin, furnishes with dimethylamine the base *dimethylaminodimethylcarbinol*, $NMe_2 \cdot CH_2 \cdot CMe_2 \cdot OH$, b. p. 160°/48 mm.; the *hydrochloride* of the *benzoyl* derivative, m. p. 202°, crystallises from absolute alcohol.

αδ-Dimethylamylene αβ-oxide, $C_5H_{11} \cdot CMe \cdot \begin{smallmatrix} CH_2 \\ | \\ O \end{smallmatrix}$, b. p. 147°/760 mm.,

D 0.8416, obtained from *αδ*-dimethylamylene chlorohydrin by the action of aqueous sodium hydroxide, yields *dimethylaminodimethyliso-amylicarbinol*, $C_5H_{11} \cdot CMe(OH) \cdot CH_2 \cdot NMe_2$, b. p. 98—99°/24 mm., on treatment with dimethylamine in benzene solution; the *hydrochloride* of the *benzoyl* derivative of this base separates in silky needles, m. p. 138°.

β -Phenylpropylene $\alpha\beta$ -oxide, $\text{CMePh} \begin{smallmatrix} \diagup \text{CH}_2 \\ \diagdown \end{smallmatrix}$, b. p. $93^\circ/33$ mm.,

D 1-0436, obtained from phenylpropylene chlorohydrin, gives rise to dimethylaminophenyldimethylcarbinol, b. p. $135\text{--}136^\circ/32$ mm., hydrochloride, needles, m. p. $159\text{--}160^\circ$, aurichloride, m. p. 110° , benzoyl derivative, oily, furnishing a hydrochloride, prisms, m. p. $205\text{--}206^\circ$.

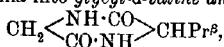
Methylaminophenyldimethylcarbinol, $\text{OH}\cdot\text{CMePh}\cdot\text{CH}_2\cdot\text{NHMe}$, b. p. $135\text{--}138^\circ/31$ mm., hydrochloride, leaflets, m. p. 153° , benzoyl and dibenzoyl derivatives both melting at 122° , was obtained from β -phenylpropylene $\alpha\beta$ -oxide and methylamine.

Methylaminodimethylethylcarbinol, $\text{OH}\cdot\text{CMeEt}\cdot\text{CH}_2\cdot\text{NHMe}$, b. p. $80^\circ/52$ mm., divaleryl derivative, oil, b. p. $162^\circ/36$ mm., results from the interaction of β -methylbutylene $\alpha\beta$ -oxide and methylamine.

G. T. M.

Synthesis of Polypeptides. XXVII. 3. Derivatives of Active Valine. EMIL FISCHER and HELMUTH SCHEIBLER (*Annalen*, 1908, 363, 136—167).—Only inactive dipeptides of valine have been prepared previously. A knowledge of the optically active forms appeared desirable, not only to permit of a comparison of the synthetical substances with the products of protein hydrolysis, but also for the study of Walden's transformation. Glycyl-*d*-valine, *d*-alanyl-*d*-valine, and *l*-leucyl-*d*-valine are readily prepared in the usual manner by coupling *d*-valine with the chlorides of the halogeno-fatty acids, and treating the products with aqueous ammonia, as are also *d*- α -bromoisovaleryl-glycine and *l*- α -bromoisovaleryl-*d*-valine by coupling *d*- and *l*- α -bromoisovaleryl chlorides with glycine and *d*-valine respectively. In order to avoid racemisation, these bromo-compounds must be converted into the dipeptides by means of anhydrous, liquid ammonia. On hydrolysis, *d*-valylglycine yields *d*-valine and glycine, whilst *l*-valyl-*d*-valine yields *dl*-valine. When treated with ammonia in methyl-alcoholic solution at 0° , the methyl ester of *l*-valyl-*d*-valine yields an inactive anhydride, termed *trans*-valine anhydride.

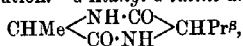
Chloroacetyl-d-valine, $\text{CH}_2\text{Cl}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^s\cdot\text{CO}_2\text{H}$, crystallises from water in prisms, m. p. $113\text{--}115^\circ$ (corr.), or from alcohol in plates; $[\alpha]_D^{20} + 15.8^\circ$. Glycyl-*d*-valine, $\text{NH}_2\cdot\text{CH}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^s\cdot\text{CO}_2\text{H}$, separates from aqueous alcohol in microscopic needles, m. p. about 254° (corr.), $[\alpha]_D^{20} - 19.7^\circ$ in aqueous, -10.5° in hydrochloric acid, and -6.9° in sodium hydroxide, solution. The hydrochloride crystallises in needles or prisms; the copper salt separates from its deep blue aqueous solution partly in microscopic prisms and partly as a vitreous mass. The hydrochloride of the methyl ester, $\text{C}_8\text{H}_{16}\text{O}_3\text{N}_2\cdot\text{HCl}$, prepared by treatment of glycyl-*d*-valine with hydrogen chloride in methyl-alcoholic solution and evaporation of the product at 25° under diminished pressure, crystallises in needles. This is converted by methyl-alcoholic ammonia into glycyl-*d*-valine anhydride,



which crystallises in needles, m. p. about 266° (corr.), $[\alpha]_D^{20} + 20.8^\circ$ in

glacial acetic acid, +32.7° in aqueous, or +41° in alcoholic, solution (Fischer, Abstr., 1907, i, 901).

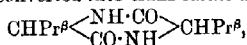
d-α-Bromopropionyl-d-valine, $\text{CHMeBr}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^{\beta}\cdot\text{CO}_2\text{H}$, prepared from *d-α*-bromopropionyl chloride and *d*-valine, forms feathery crystals, m. p. 180° (corr.), $[\alpha]_D^{20} + 20.8^\circ$ to +21° in alcoholic solution. *d-Alanyl-d-valine*, $\text{NH}_2\cdot\text{CHMe}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^{\beta}\cdot\text{CO}_2\text{H}$, crystallises from dilute alcohol in microscopic needles, m. p. about 265° (corr.), $[\alpha]_D^{20} - 1.9^\circ$ in hydrochloric acid, -4.5° in *N*-sodium hydroxide, or -5.4° in aqueous, solution. *d-Alanyl-d-valine anhydride*,



is prepared by conversion of the dipeptide into its *methyl ester hydrochloride*, and treatment of this with ammonia in methyl-alcoholic solution; it crystallises in needles, m. p. about 268—270° (corr.), $[\alpha]_D^{20} - 29.3^\circ$ in glacial acetic acid solution, and has less inclination than the preceding anhydride to separate from its solutions in the gelatinous state.

d-α-Bromoisohexoyl-d-valine, $\text{CH}_2\text{Pr}^{\beta}\cdot\text{CHBr}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^{\beta}\cdot\text{CO}_2\text{H}$, prepared from *d-α*-bromoisohexoyl chloride and *d*-valine, forms stout crystals, m. p. 150—151°, $[\alpha]_D^{20} + 24.3^\circ$ in alcoholic solution. *l-Leucyl-d-valine*, $\text{NH}_2\cdot\text{CH}(\text{C}_4\text{H}_9)\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^{\beta}\cdot\text{CO}_2\text{H}$, crystallises from aqueous alcohol in spears or prisms containing water of crystallisation, which is lost in a vacuum over sulphuric acid, m. p. about 282° (corr.; slight decomp.) when quickly heated, $[\alpha]_D^{20} + 18.0^\circ$ in aqueous solution. The *anhydride*, $\text{C}_{11}\text{H}_{20}\text{O}_2\text{N}_2$, formed by way of the *methyl ester*, crystallises in microscopic needles, m. p. about 282°, $[\alpha]_D^{20} - 46.5^\circ$ to -50.2° in glacial acetic acid solution.

l-α-Bromoisovaleryl-d-valine, $\text{CHPr}^{\beta}\text{Br}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHPr}^{\beta}\cdot\text{CO}_2\text{H}$, prepared from *l-α*-bromoisovaleryl chloride and *d*-valine, crystallises in needles, m. p. 163—165° (corr.), $[\alpha]_D^{20} - 22.7^\circ$ in absolute alcohol. *l-Valyl-d-valine*, $\text{C}_{10}\text{H}_{20}\text{O}_2\text{N}_2\cdot 1\frac{1}{2}\text{H}_2\text{O}$, crystallises in needles, and loses the water of crystallisation at 95°/12—15 mm. over phosphoric oxide, m. p. about 308° (corr.), $[\alpha]_D^{20} - 70.6^\circ$ to -74.0° . The *hydrochloride* forms quadratic crystals, and is readily soluble in water. The *hydrochloride* of the methyl ester, formed in the usual manner, crystallises in needles, and on treatment with ammonia in methyl-alcoholic solution is converted into *trans-valine anhydride*,



which crystallises in prisms, m. p. 316—318° (corr.), and is optically inactive.

d-Valylglycine, $\text{NH}_2\cdot\text{CHPr}^{\beta}\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, prepared from *d-α*-bromoisovaleryl-glycine (Fischer and Scheibler, this vol., i, 858) by the action of liquid ammonia at 25°, crystallises from aqueous alcoholic-etheral solution in short prisms, m. p. about 272° (corr.), $[\alpha]_D^{20} + 89.8^\circ$ to +93.6° in aqueous solution, or $[\alpha]_D^{20} + 39.4^\circ$ in 10% hydrochloric acid. A by-product of the formation of this dipeptide crystallises on evaporation of the alcoholic-etheral mother liquor in prisms, and has an acid reaction and taste. G. Y.

Distillation of Creatinine. R. ENGELAND (*Zeitsch. physiol. Chem.*, 1908, 57, 65—66).—The following compounds have been isolated from

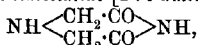
the products obtained by the destructive distillation of creatinine hydrochloride: ammonia, dimethylamine, and pyrrole. J. J. S.

Glycocholic Acid. MAURICE PIETTRE (*Compt. rend.*, 1908, 147, 810—813).—A convenient method is described for isolating glycocholic acid from pig's bile. The product amounts to 60—75% of the total weight of glycocholic acid present, and is quite free from taurocholic acid; its composition agrees with the formula $C_{27}H_{45}O_6N$. The pure acid has m. p. 150° , and $[\alpha]_D + 21^\circ 54'$ in alcoholic solution.

Alkali hydroxides have no action on glycocholic acid under ordinary conditions, but under pressure, complicated changes, exceeding the limits of simple hydrolysis, occur. The toxic properties of sodium glycocholate have been determined; with the higher animals, the lethal dose varies according to the method of administration. W. O. W.

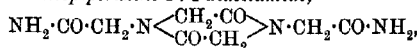
Iminodiacetic Acid and some Derivatives. W. J. A. JONGKEES (*Rec. trav. chim.*, 1908, 27, 287—326).—The derivatives of iminodiacetic acid, $NH(CH_2 \cdot CO_2H)_2$ (diglycollamidic acid, iminoacetic acid), described in the original can be divided into three groups: (I) derivatives containing the imino-group; (II) derivatives in which the hydrogen of the imino-group is replaced by acyl radicles, (III) hydantoin derivatives.

(I). The *hydrochloride* of iminodiacetic acid, $HCl \cdot NH(CH_2 \cdot CO_2H)_2$, obtained by the action of hydrogen chloride on the acid in the presence of alcohol, decomposes at 255° , and is unchanged by the action of such dehydrating agents as thionyl chloride or phosphorus pentachloride; the *ethyl ester*, $HCl \cdot NH(CH_2 \cdot CO_2Et)_2 \cdot H_2O$, has m. p. 74° , and, on treatment with alcoholic ammonia, yields *ethyl hydrogen iminodiacetate*, $NH(CH_2 \cdot CO_2H) \cdot CH_2 \cdot CO_2Et$, m. p. 175 — 176° ; the *hydrochloride*, $HCl \cdot NH(CH_2 \cdot CO_2H) \cdot CH_2 \cdot CO_2Et$, has m. p. 143° ; the *methyl ester*, $HCl \cdot NH(CH_2 \cdot CO_2Me)_2$, has m. p. 183° , and yields *methyl iminodiacetate*, $NH(CH_2 \cdot CO_2Me)_2$, b. p. $126^\circ/33$ mm. or $123.5^\circ/16$ mm., $D_{20}^{25} 1.1675$, from which the diamide, $NH(CH_2 \cdot CO \cdot NH_2)_2$, m. p. 143° (Heintz, *Annalen*, 1868, 148, 178), and the *monamide* (*iminoacetamic acid*), $NH(CH_2 \cdot CO_2H \cdot CH_2 \cdot CO \cdot NH_2)$, m. p. 210° , are obtained by the action of ammonia; the *hydrochlorides* have m. p. 255° and 210° respectively; and the *compound*, $NH_3 \cdot HCl \cdot NH(CH_2 \cdot CO \cdot NH_2)_2$, obtained by treating the hydrochloride of methyl iminodiacetate with methyl-alcoholic ammonia, forms beautiful crystals, that soften at 145° , but do not melt at 190° . *Iminodiacetamide* [$2:6$ -diketopiperazine],

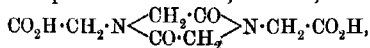


obtained by heating under 15 mm. pressure the ammonium salt, the monamide, or the diamide of the acid, forms white needles, m. p. 200 — 205° .

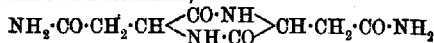
(II). *2:5-Diketopiperazine-1:4-diacetamide*,



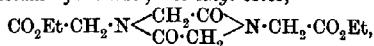
is obtained as a secondary product in the preparation of iminodiacetamide; it decomposes at 303 — 305° ; the *acid*,



forms crystals, m. p. 280—290°, and, unlike the isomeride, 2:5-di-ketopiperazine-3:6-diacetamide,

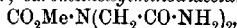


(Fischer, Abstr., 1905; i, 32), is stable towards boiling methyl-alcoholic potassium hydroxide; the *ethyl* ester,



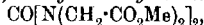
has m. p. 79—80°. *Acetylminodiacyetonitrile*, $\text{NAc}(\text{CH}_2\cdot\text{CN})_2$, obtained by the action of acetic anhydride on iminodiacetonitrile, is a viscid oil, b. p. 227°/16 mm.; *methyl acetylminodiacyetate*, $\text{NAc}(\text{CH}_2\cdot\text{CO}_2\text{Me})_2$, similarly prepared, has b. p. 184—185°/15 mm., m. p. 83.5—84°; *acetylminodiacyetamide*, $\text{NAc}(\text{CH}_2\cdot\text{CO}\cdot\text{NH}_2)_2$, has m. p. 203°, and yields the *hydrochloride*, $\text{HCl}\cdot\text{NAc}(\text{CH}_2\cdot\text{CO}\cdot\text{NH}_2)_2$.

Carbmethoxyiminodiacyetonitrile, $\text{CO}_2\text{Me}\cdot\text{N}(\text{CH}_2\cdot\text{CN})_2$, prepared from methyl chloroformate and iminodiacetonitrile, has m. p. 63—64°, b. p. 189°/15 mm.; *methyl carbmethoxyiminodiacyetate*, $\text{CO}_2\text{Me}\cdot\text{N}(\text{CH}_2\cdot\text{CO}_2\text{Me})_2$, has b. p. 167°/15 mm.; *carbmethoxyiminodiacyetamide*,

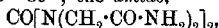


has m. p. 212°; *carbmethoxyiminodiacyetic acid*, $\text{CO}_2\text{Me}\cdot\text{N}(\text{CH}_2\cdot\text{CO}_2\text{H})_2$, has m. p. 123°, and the blue copper salt, $\text{C}_6\text{H}_7\text{O}_6\text{NCu}\cdot\text{H}_2\text{O}$, and the barium salt, $\text{C}_6\text{H}_7\text{O}_6\text{NBA}_2\cdot\text{H}_2\text{O}$, have been prepared.

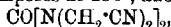
Methyl chloroformyliminodiacyetate, $\text{COCl}\cdot\text{N}(\text{CH}_2\cdot\text{CO}_2\text{Me})_2$, obtained by the action of carbonyl chloride (1 mol.) on methyl iminodiacyetate (2 mols.), has m. p. 74°; *methyl carbonyldi-iminodiacyetate*,



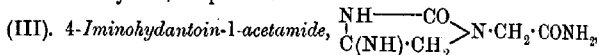
from carbonyl chloride (1 mol.) and methyl iminodiacyetate (4 mols.), forms needles, m. p. 88—89°; the *amide*,



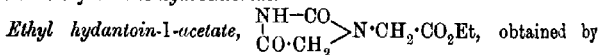
darkens at 230° and decomposes at 250°, and the *nitrile*,



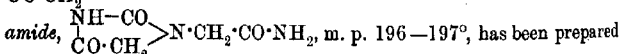
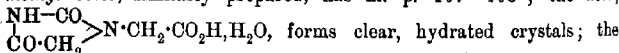
forms white crystals, m. p. 155°.



obtained by the action of alcoholic ammonia on carbmethoxyimino-diacyetonitrile, forms colourless needles, which decompose at 245°, and yields a crystalline *hydrochloride*.



hydrolysis of the preceding compound with hot hydrochloric acid and subsequent esterification, forms beautiful needles, m. p. 84—85°; the *methyl* ester, similarly prepared, has m. p. 107—108°; the *acid*,

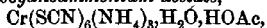


(1) by the action of alcoholic ammonia on methyl chloroformyliminodiacyetate; (2) as a secondary product in the hydrolysis of imino-hydantoin-1-*acetamide*, and (3) by the action of ammonia on the *ethyl* ester.

M. A. W.

Hexathiocyano-salts of Molybdenum. JOHANNA MAAS and JULIUS SAND (*Ber.*, 1908, 41, 3367—3376. Compare this vol., i, 397, 513).—A reply to the criticism of Rosenheim and Garfunkel (this vol., i, 614). The authors have carefully re-analysed the potassium, ammonium, and zinc amino-salts, and confirm their previous results.

The *chromihexathiocyanoammonium acetate*,



prepared from the chloride, $\text{CrCl}_2(\text{H}_2\text{O})_4\text{Cl} \cdot 2\text{H}_2\text{O}$, ammonium thiocyanate, and acetic acid, crystallises in reddish-violet, rhombic needles, isomorphous with the corresponding molybdenum salt. W. R.

Preparation of Additive Compounds of Chloral with Amides. NATHAN SULZBERGER (D.R.-P. 198715).—Chloral combines additively with the amides of the higher fatty acids. *Chloral palmitinamide*, $\text{CCl}_3 \cdot \text{CH}(\text{OH}) \cdot \text{NH} \cdot \text{CO} \cdot [\text{CH}_2]_{14} \cdot \text{CH}_3$, an oleaginous, white, crystalline powder, m. p. 110° , is produced by mixing its generators and warming at 100° . *Chloral α -bromopalmitin anilide*, prepared in a similar manner, is a colourless, crystalline powder with a greasy feel.

G. T. M.

Action of Phosphorus Pentachloride on Halogenated Acid Amides. WILHELM STEINKOPF (*Ber.*, 1908, 41, 3571—3595).—Although in regard to their rates of formation and stability the halogen-substituted acetonitriles and acetamides closely resemble nitroacetamide and -acetonitrile, it is found that they do not yield the corresponding acetimide chlorides when treated with thionyl chloride, whereas, as shown by Steinkopf and Bohrmann (this vol., i, 327), the action of thionyl chloride on nitroacetamides leads to the formation of nitroacetimide chlorides. The author has studied therefore the action of phosphorus pentachloride on dichloro- and dibromoacetonitriles.

Wallach found (this Journ., 1877, ii, 182) that, whilst the action of phosphorus pentachloride on acetamide leads to the formation of the amide chloride, which readily loses hydrogen chloride, forming the imide chloride, di- and tri-chloroacetamides yield products which may have the formulæ (1) $\text{CHCl}_2 \cdot \text{CO} \cdot \text{NCl} \cdot \text{PCl}_2$ and $\text{CCl}_3 \cdot \text{CO} \cdot \text{NCl} \cdot \text{PCl}_2$ or (2) $\text{CHCl}_2 \cdot \text{CCl} \cdot \text{N} \cdot \text{POCl}_2$ and $\text{CCl}_3 \cdot \text{CCl} \cdot \text{N} \cdot \text{POCl}_2$ respectively. It is found now that the formation of such phosphorus compounds is a general reaction of the α -halogenated acetamides. The formulæ (2) are to be preferred, as the action of moisture on these compounds leads to the displacement of a chlorine atom by a hydroxyl group, and the formation of a product, $\text{CHCl}_2 \cdot \text{C}(\text{OH}) \cdot \text{N} \cdot \text{POCl}_2$, or its tautomeric form, $\text{CHCl}_2 \cdot \text{CO} \cdot \text{NH} \cdot \text{POCl}_2$, is more probable than that of a product, $\text{CHCl}_2 \cdot \text{CO} \cdot \text{N}(\text{OH}) \cdot \text{PCl}_2$.

[With CZESLAU BENEDEK.]—The liquid product, obtained by the action of phosphorus pentachloride on chloroacetamide in absence of moisture (Wallach, *loc. cit.*), gives, on analysis, figures agreeing with the formula $\text{CH}_2\text{Cl} \cdot \text{CCl} \cdot \text{N} \cdot \text{POCl}_2$; it decomposes on distillation in a vacuum, and, on exposure to moisture, evolves hydrogen chloride and yields small amounts of crystalline chloroacetylphosphamic chloride, $\text{CH}_2\text{Cl} \cdot \text{CO} \cdot \text{NH} \cdot \text{POCl}_2$.

Dichloroacetylphosphamic chloride, $\text{CHCl}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{POCl}_2$, formed by the action of moist air on $\alpha\beta$ -trichloroethylidenephosphamic chloride (Wallach, *loc. cit.*), crystallises in white plates, m. p. 112—113°. When treated with sodium ethoxide in alcoholic solution, it forms the *ethyl* ester, $\text{CHCl}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{OEt})_2$, which crystallises in white needles, m. p. 72—73°, is soluble in dilute sodium hydroxide or ammonia, forms a yellow *precipitate* with platinum tetrachloride in ammoniacal solution, and can be titrated with *N*/10 potassium hydroxide. The *dianilide*, $\text{CHCl}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{NHPh})_2$, prepared by heating the oxydichloride with aniline in ethereal solution, or by the successive action of an excess of aniline and of water on $\alpha\beta$ -trichloroethylidenephosphamic chloride, crystallises from alcohol in white needles, m. p. 219—220°. The *bisphenylhydrazide*, $\text{CHCl}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{NH}\cdot\text{NHPh})_2$, forms white crystals, m. p. 190° (decomp.).

Trichloroacetylphosphamic chloride, $\text{CCl}_3\cdot\text{CO}\cdot\text{NH}\cdot\text{POCl}_2$, prepared by evaporating the light petroleum solution of the imide chloride-phosphorus oxydichloride (Wallach, *loc. cit.*) in contact with moist air, crystallises in white needles, m. p. 146—148°, and when treated with water yields phosphoric acid and trichloroacetamide. The *methyl* ester, $\text{CCl}_3\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{OMe})_2$, crystallises in white leaflets, m. p. 105—107°, and dissolves in dilute alkalis; the *potassium* salt, $\text{C}_2\text{H}_5\text{O}_2\text{NCl}_3\text{PK}$, m. p. about 135°, is hygroscopic. The *ethyl* ester, $\text{C}_2\text{H}_5\text{O}_2\text{NCl}_3\text{P}$, forms crystalline aggregates, m. p. 47—48°. The *dianilide*, $\text{C}_{14}\text{H}_{13}\text{O}_2\text{N}_3\text{Cl}_3\text{P}$, white needles, m. p. 194—195°. The *bisphenylhydrazide*, $\text{C}_{14}\text{H}_{13}\text{O}_2\text{N}_5\text{Cl}_3\text{P}$, m. p. 237—238° (decomp.).

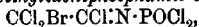
The product of the action of phosphorus pentachloride on bromoacetamide is unstable and has not been isolated.

[With H. GRÜNUPF.]—*Tribromochloroethylidenephosphamic chloride*, $\text{CBr}_3\cdot\text{CCl}\cdot\text{N}\cdot\text{POCl}_2$, formed from phosphorus pentachloride and tribromoacetamide at 90—110°, is obtained as a liquid, which gradually crystallises. *Tribromoacetylphosphamic chloride*, $\text{CBr}_3\cdot\text{CO}\cdot\text{NH}\cdot\text{POCl}_2$, formed by exposing the preceding substance to air, separates from benzene in crystals, m. p. 105—106°, and decomposes on further exposure to moisture. When treated with sodium methoxide in methyl-alcoholic solution, it forms *methyl dibromomethoxyacetylphosphamate*,
 $\text{OMe}\cdot\text{CBr}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{OMe})_2$,

which separates from water in white crystals, m. p. 92—93°. The corresponding *ethoxy-ethyl* ester, $\text{C}_8\text{H}_{16}\text{O}_5\text{NBr}_2\text{P}$, forms long needles, m. p. 91°.

[With G. KIRCHHOFF.]— $\alpha\beta$ -*Dichloro- β -bromoethylidenephosphamic chloride*, $\text{CHClBr}\cdot\text{CCl}\cdot\text{N}\cdot\text{POCl}_2$, prepared from chlorobromoacetamide compound, is obtained as a yellow liquid. The *amide*, formed by the action of moisture on this, could not be purified, and was converted directly into the *ethyl* ester, $\text{CHClBr}\cdot\text{CO}\cdot\text{NH}\cdot\text{PO}(\text{OEt})_2$, colourless crystals, m. p. 67—68°.

$\alpha\beta$ -*Trichloro- β -bromoethylidenephosphamic chloride*,



prepared from dichlorobromoacetamide, forms crystals, m. p. 68°. The *chloride*, $\text{CCl}_2\text{Br}\cdot\text{CO}\cdot\text{NH}\cdot\text{POCl}_2$, forms colourless crystals, m. p. 147°;

the *methyl* ester of this, $C_4H_5O_4NCl_2BrP$, separates from water in crystals, m. p. 107°; the *ethyl* ester, m. p. 76—77°.

Dichloronitroacetamide, $NO_2 \cdot CCl_2 \cdot CO \cdot NH_2$, prepared by the action of chlorine on ammonio-nitroacetamide, is obtained as a white, crystalline precipitate, m. p. 94—95°. The $\alpha\beta\beta$ -trichloro- β -nitro-phosphamic chloride, $NO_2 \cdot CCl_2 \cdot CCl : N \cdot POCl_2$, forms white crystals, m. p. 55—60°. *Dichloronitroacetylphosphamic chloride*, $C_2HO_4N_2Cl_2P$, separates from benzene in crystals, m. p. 165° (decomp.); the *ethyl* ester, $C_6H_{11}O_6N_2Cl_2P$, long needles, m. p. 56°.

α -Chloro- $\beta\beta$ -dibromo- β -nitroacetylphosphamic chloride,
 $NO_2 \cdot CBr_2 \cdot CCl : N \cdot POCl_2$,

forms white crystals, m. p. about 65°. *Dibromonitroacetylphosphamic chloride*, $C_2HO_4N_2Cl_2Br_2P$, white crystals, m. p. 187—188° (decomp.).

[With C. BENEDEK.]— α -Chloro- β -bromophenylethylidenephosphamic chloride, $CHBrPh \cdot CCl : N \cdot POCl_2$, was analysed. The derivatives are unstable.

α -Chlorodiphenylacetamide has m. p. 111—113° (115°: Bickel, Abstr., 1889, 999). *Chlorodiphenylacetylphosphamic chloride*,

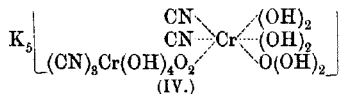
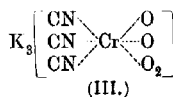
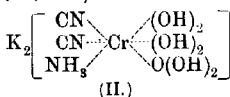
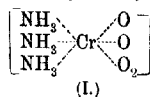
$ClPh_2 \cdot CO \cdot NH \cdot POCl_2$,

forms crystals resembling ammonium chloride, m. p. 122—123°. The *methyl* ester, $C_{10}H_{17}O_4NClP$, white crystals, m. p. 104—106°.

$\alpha\beta\beta$ -Trichloropropylidenephosphamic chloride, $ClMeCl_2 \cdot CCl : N \cdot POCl_2$, prepared from di- α -chloropropionamide and phosphorus pentachloride at 85—95°, crystallises from light petroleum in white needles, m. p. 80°. *Di- α -chloropropionylphosphamic chloride*, $C_3H_4O_2NCl_2P$, snow-white needles, m. p. 127—128°.

Other nitro-amides, such as nitroacetamide and nitromalonamide, also react with phosphorus pentachloride, but the products are too unstable to permit of their isolation. G. Y.

Higher Oxidation Products of Chromium. IV. Chromium Tetroxide Compounds. ERNST H. RIESENFELD [and, in part, ALFRED WESCH] (*Ber.*, 1908, 41, 3536—3552. Compare Abstr., 1906, ii, 92; this vol., ii, 951; Riesenfeld and Wohlers, Abstr., 1907, ii, 357).—The mol. wt. of Wiede's triamminechromium tetroxide (Abstr., 1898, ii, 28) has been determined by the cryoscopic method, and the values obtained found to agree with the formula $(NH_3)_3CrO_4$ (compare Werner, Abstr., 1906, ii, 760). The structural formula I has been

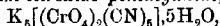


assigned to this compound; in support of this formula, it has been found possible to prepare the dicyanommonamine derivative, II, inter-

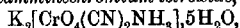
mediate between the tricyano-compound, III, of Wiede (*loc. cit.*) and the triamminechromium tetroxide. A compound has also been prepared having the formula IV, in which one of the positions in the complex is occupied by a group similar in composition to the rest of the complex radicle. This is probably the first complex salt of this nature to be prepared.

The complex in II and IV is represented as containing water, since compound II crystallises with water, which is removed together with ammonia when the substance is kept over calcium chloride, and compound IV crystallises with water, only part of which is eliminated over phosphoric oxide. One mol. of the triammine, when decomposed by acid or alkali, yields 2—3 equivalents of hydrogen peroxide, that is, only the O_2 group in the complex forms hydrogen peroxide, and is thus in all probability a bivalent group. It is therefore highly probable that the chromium in the tetroxide derivatives is sexavalent.

Potassium dichromium tetroxide pentacyanide,



is formed by treating an aqueous solution of potassium cyanide and chromic acid, cooled in an ice-salt freezing mixture, with hydrogen peroxide; it crystallises in dark reddish-brown prisms. Molecular-weight determinations by the cryoscopic method and electrical conductivity measurements show that the salt when completely dissociated in aqueous solution is ionised, thus: $K_5[(CrO_4)_2(CN)_5] = 5K^+ + [(CrO_4)_2CN]^- + 4CN^-$. It is converted by aqueous ammonia into *potassium dicyanommoniamminechromium tetroxide,*



crystallising in long, slender, yellowish-brown needles. The complex of this salt is dissociated in dilute aqueous solution. It slowly evolves hydrogen cyanide and ammonia when kept, and decomposes with slight explosion when heated rapidly.

W. H. G.

Preparation of the Cyanides and Cyanamides of the Alkali and Alkaline Earth Metals. BADISCHE ANILIN- & SODA FABRIK (D.R.-P. 200986).—All nitrogen compounds of titanium, such as the nitride or the carbonitrides, readily give rise to cyanides or cyanamides when heated with carbon and the oxides, carbonates, sulphates, or other salts of the alkali and alkaline earth metals. The reaction occurs either in the presence or absence of a fusible medium, such as fusion mixture or a fusible chloride. The carbon may be introduced in the form of soot or pitch, or it may be obtained from a decomposable hydrocarbon. The titanium is left in a form capable of recombining with carbon, and thus the process is continuous. The alkali metals give rise to cyanides only; barium yields a mixture of cyanide and cyanamide, whereas calcium cyanamide is the sole product when lime is employed.

G. T. M.

Preparation of Aminodicyanodiamidine. TEMISTOCLE JONA (*Gazzetta*, 1908, 38, ii, 480—484).—Thiele and Uhlfelder's method of preparing aminodicyanodiamidine by nitrating dicyanodiamidine and subsequently reducing (Abstr., 1899, i, 119) gives low yields. As much as 80% of the theoretical quantity can be obtained by reducing nitrodicyanodiamidine by means of hydrochloric acid (D 1.19) and

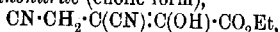
zinc dust at a temperature not higher than 45°. When aminodicyanodiamidine dihydrochloride is heated with *p*-aminobenzaldehyde in a solution faintly acid by hydrochloric acid, it yields a red compound, crystallising from alcohol in long filaments and softening at 300° without showing signs of melting; the constitution of this compound is being investigated.

T. H. P.

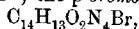
Desmotropy and Fluorescence of Ethyl Oxalosuccinonitrile. WILHELM WISLICENUS and PAUL BERG (*Ber.*, 1908, 41, 3757—3768).—Ethyl oxalate (1 mol.) and succinonitrile (1 mol.) condense under the influence of sodium ethoxide, yielding the sodium derivative of ethyl oxalosuccinonitrile, $\text{CN}\cdot\text{CH}_2\cdot\text{CH}(\text{CN})\cdot\text{CO}\cdot\text{CO}_2\text{Et}$. Attempts to bring about condensation with a second molecule of ethyl oxalate were unsuccessful, although succinonitrile condenses with 2 mols. of methyl oxalate; the methyl analogue of the ethyl compound mentioned above could not, however, be prepared. Ethyl oxalosuccinonitrile exists in two tautomeric forms; the enolic form is obtained by treating the sodium derivative with acid, and passes into the ketonic form when warmed in aqueous solution. The ketonic form dissolves in alcohol, forming a bluish-violet, fluorescent solution. This is the first fluorescent substance of the aliphatic series to be prepared, and is consequently of some importance in connexion with the question of the relationship between chemical constitution and fluorescence.

The chemical behaviour of the ketone is represented by the formula $\text{CN}\cdot\text{CH}_2\cdot\text{CH}(\text{CN})\cdot\text{CO}\cdot\text{CO}_2\text{Et}$ with but one exception, namely, its behaviour towards phenylhydrazine; although the enolic form yields a normal phenylhydrazone, the ketonic form gives rise to only small quantities of indefinite substances.

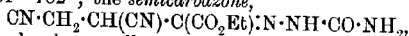
α -Ethyl oxalosuccinonitrile (enolic form),



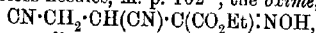
crystallises in colourless, pointed leaflets, m. p. 102—103°. The alcoholic solution is not fluorescent, and gives a dark cherry-red coloration with ferric chloride; the copper salt, $(\text{C}_8\text{H}_7\text{O}_3\text{N}_2)_2\text{Cu}$, forms emerald-green, rhombic leaflets, m. p. 118—121° (decomp.). It yields with phenylcarbimide at the ordinary temperature the additive product, $\text{CN}\cdot\text{CH}_2\cdot\text{C}(\text{CN})\cdot\text{C}(\text{O}\cdot\text{CO}\cdot\text{NHPh})\cdot\text{CO}_2\text{Et}$, small, flat needles, decomposing at 200°; with ammonia in ethereal solution, the ammonium salt, $\text{C}_8\text{H}_{11}\text{O}_3\text{N}_3$, long leaflets, decomposing at 145—150°; with phenylhydrazine in alcoholic solution, the phenylhydrazone, $\text{C}_{14}\text{H}_{14}\text{O}_2\text{N}_4$, colourless needles, m. p. 150—151°; the *p*-bromophenylhydrazone,



has m. p. 181—182°; the semicarbazone,



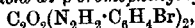
forms long, colourless needles, m. p. 162°; the oxime,



crystallises in long needles, m. p. 110°.

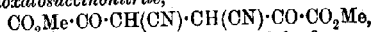
β -Ethyl oxalosuccinonitrile (ketonic form) crystallises in pale yellow, microscopic needles, m. p. 154—155°; it is reconverted into the α -form by an alcoholic solution of sodium ethoxide. It does not react with ammonia, *p*-bromophenylhydrazine, semicarbazide hydrochloride, hydroxylamine, or phenylcarbimide at the ordinary tempera-

ture, and yields with phenylhydrazine very small quantities of a substance, which crystallises in small, golden-yellow needles, decomposing at 122—125°. It reacts with phenylcarbimide at 100°, yielding the additive product described above, and with *p*-bromophenylhydrazine at 100°, forming *oxalic acid di-p-bromophenylhydrazide*,



colourless scales, m. p. 240° (decomp.).

Dimethyl dioxalosuccinonitrile,



crystallises in yellow, glistening, hexagonal leaflets, m. p. 185°. It does not give a coloration with ferric chloride, but with copper acetate forms a copper salt, $(\text{C}_{10}\text{H}_7\text{O}_6\text{N}_2)_2\text{Cu}$, crystallising in yellowish-green, microscopic needles.

W. H. G.

Negative Substituted Amino-oximes. III. Brominated Amino-oximes. WILHELM STEINKOPF and H. GRÜNUPP (*Ber.*, 1908, 41, 3569—3571).—The authors have prepared the mono-, di-, and tri-bromoethenylamino-oximes, and have found that, whilst the mono- and di-bromo-compounds resemble in their stability the chloro- and iodo-substituted ethenylamino-oximes, the tribromo-compound is comparatively unstable. The brominated ethenylamino-oximes are prepared by the action of hydroxylamine on the corresponding brominated acetonitriles in the same manner as are the chloro- and iodo-compounds. They give characteristic brown to brownish-red colorations with ferric chloride, and form hydrochlorides when treated with hydrogen chloride in ethereal solution.

Bromoethenylamino-oxime, $\text{CH}_2\text{Br}\cdot\text{C}(\text{NOH})\cdot\text{NH}_2$, crystallises in yellowish-white leaflets, m. p. 95—96°, yields a dirty-green precipitate with alkaline copper sulphate solution, and reduces boiling alkaline mercuric chloride.

Dibromoethenylamino-oxime, $\text{CHBr}_2\cdot\text{C}(\text{NOH})\cdot\text{NH}_2$, crystallises in white needles, m. p. 120°, forms a dirty-green precipitate with alkaline copper sulphate, and reduces mercuric chloride in boiling alkaline solution. The *hydrochloride*, white powder, m. p. 163—165° (decomp.).

Tribromoethenylamino-oxime, $\text{CBr}_3\cdot\text{C}(\text{NOH})\cdot\text{NH}_2$, m. p. 126°, gradually decomposes at the ordinary temperature.

G. Y.

Silicoiodoform. OTTO RUFF [in part, EMIL GEISEL] (*Ber.*, 1908, 41, 3738—3744).—Silicoiodoform may be obtained in fairly large quantities by the action of hydrogen iodide on siliconitrogen hydride, SiNH , suspended in cold carbon disulphide, or more readily by treating trianilinosilicon hydride, $\text{SiH}(\text{NHPh})_3$, with hydrogen iodide in benzene.

Silicoiodoform decomposes slowly above 150°, liberating hydrogen and probably a volatile iodosilicon hydride. The b. p. of silicoiodoform is about 220°/760 mm., but on continued boiling the temperature slowly rises to about 300°; the residue left after distillation is chiefly silicon tetraiodide. Silicoiodoform boils without decomposition under a pressure of 14, 22, 67, and 122 mm. at 106°, 111°, 132°, and 155° respectively; it has D^{25}_D 3.286.

Trianilinosilicon hydride (trianilinosilicane), $\text{SiH}(\text{NHPh})_3$, prepared

by the interaction of aniline and silicochloroform in benzene, crystallises in slender, white needles, and begins to decompose at 114° . It is converted by hydrogen chloride, bromide, or iodide into the corresponding trihalogenated silicane, and by water into silicoformic acid and aniline.

Siliconitrogen hydride, SiNH , is obtained together with siliconamide by the interaction of silicochloroform vapour and ammonia diluted with hydrogen at about -10° , as a white powder. W. H. G.

Silico-acids and their Derivatives. WILHELM MELZER (*Ber.*, 1908, 41, 3390—3395).—Silicon tetrachloride reacts with organo-magnesium halides, yielding compounds of the type RSiCl_3 (compare Kipping, *Trans.*, 1907, 91, 209), alkyltrichlorosilanes. These react with water, yielding the silico-acids, $\text{R}\cdot\text{SiO}_2\text{H}$, and with anhydrous alcohol, yielding the ortho-esters, $\text{R}\cdot\text{Si}(\text{OEt})_3$. In the preparation of the chlorides, it is essential that all traces of moisture should be removed.

Propyltrichlorosilane, $\text{C}_3\text{H}_7\cdot\text{SiCl}_3$, is a clear, colourless liquid, b. p. $123-125^{\circ}$; it has a penetrating odour, and fumes in contact with the air.

Silicobutyric acid, $\text{C}_3\text{H}_7\cdot\text{SiO}_2\text{H}$, forms a hard, solid mass, which can be ground to a powder. It is infusible.

Ethyl orthosilicobutyrate, $\text{C}_3\text{H}_7\cdot\text{Si}(\text{OEt})_3$, is a colourless liquid with an aromatic odour, and has b. p. $177-179^{\circ}$ and D 0.8945 .

isoAmyltrichlorosilane, $\text{C}_5\text{H}_{11}\cdot\text{SiCl}_3$, is a colourless liquid, b. p. 46.9° mm., D 1.066 .

Ethyl orthosilicohexoate, $\text{C}_5\text{H}_{11}\cdot\text{Si}(\text{OEt})_3$, has b. p. $195-200^{\circ}$ and D 0.9318 .

Silicohexoic acid, $\text{C}_5\text{H}_{11}\cdot\text{SiO}_2\text{H}$, forms a hard, brittle solid, soluble in ether, benzene, or chloroform, and has not a definite m. p.

Benzyltrichlorosilane, $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{SiCl}_3$, has b. p. $94-96^{\circ}/11$ mm. and D 1.2834 . The ortho-ester, $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{Si}(\text{OEt})_3$, has b. p. $245-250^{\circ}$ and D 0.9864 . *Silicophenylacetic acid*, $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{SiO}_2\text{H}$, separates from ether as a clear, vitreous solid, m. p. $65-66^{\circ}$.

a-Naphthyltrichlorosilane, $\text{C}_{10}\text{H}_7\cdot\text{SiCl}_3$, is a viscid liquid with a penetrating odour, and has b. p. $165-170^{\circ}/22$ mm. and D 1.3760 . The ortho-ester, $\text{C}_{10}\text{H}_7\cdot\text{Si}(\text{OEt})_3$, has b. p. $220-230^{\circ}/18$ mm., and the acid, $\text{C}_{10}\text{H}_7\cdot\text{SiO}_2\text{H}$, has m. p. $125-130^{\circ}$ (compare Khotinsky and Seregenkoff, this vol., i, 1032).

J. J. S.

Velocity of Decomposition of the Ozonides of Certain Cyclic Hydrocarbons. CARL D. HARRIES and HANS VON SPLAWA NEYMANN (*Ber.*, 1908, 41, 3552—3558).—In the communication of Harries and Neresheimer (*Abstr.*, 1906, i, 833), it is incorrectly stated that a sparingly soluble ozonide of cyclohexene is obtained by passing ozone into a solution of the hydrocarbon in chloroform. This ozonide is formed when carbon tetrachloride is the solvent, whilst when chloroform or hexane is the solvent employed, an ozonide is obtained which may be crystallised from alcohol. The two ozonides have approximately the same composition, and behave similarly when decomposed by water.

The decomposition of the ozonides of cyclopentene and cyclohexene

by water has been studied quantitatively (compare Harries and Tank, this vol., i, 517); it is found that under similar conditions *cyclopentene* ozonide is decomposed in one hour to about the same extent as *cyclohexene* ozonide in two and a-half hours.

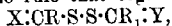
α-cycloHexene ozonide, $C_6H_{10}O_3$, obtained by passing ozone into a solution of the unsaturated hydrocarbon in hexane, crystallises in stellate groups of small, white needles, m. p. 75° ; it decomposes at 90° .

β-cycloHexene ozonide is formed together with the *α-ozonide* by acting on a solution of the hydrocarbon in carbon tetrachloride with ozone; it is a sparingly soluble, solid substance, m. p. $115-120^\circ$ (decomp.), and when analysed gives values which lie between those required for $C_6H_{10}O_3$ and $C_6H_{10}O_4$.

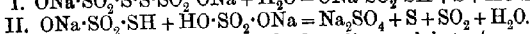
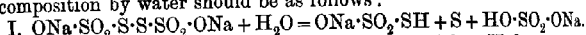
Both ozonides are decomposed by boiling water, yielding adipic acid (about 44%), adipic dialdehyde, *cyclopentenealdehyde*, and adipic semialdehyde (about 13%). *Adipic semialdehyde* (*δ-aldehydovaleric acid*), $CHO \cdot [CH_2]_4 \cdot CO_2H$, forms colourless crystals, m. p. $124-125^\circ$; the *p-nitrophenylhydrazone* crystallises in yellow needles, m. p. 134° .

W. H. G.

Lowest Oxides of Hydrogen Sulphide. EMIL FROMM (*Ber.*, 1908, 41, 3397-3425).—[With ADOLF ROESICKE.]—In previous communications (Abstr., 1906, i, 656; 1907, i, 982; this vol., i, 700), the author has stated the rule that organic disulphides of the type



containing neighbouring double linkings, are decomposed by water or alkalis, sulphur being eliminated. An attempt has been made to apply the rule to the elucidation of the constitution of sodium tetrathionate. If this salt be represented as $ONa \cdot SO_2 \cdot S \cdot S \cdot SO_2 \cdot ONa$, its decomposition by water should be as follows:



The amounts of sulphur and of sodium sulphate (compare Guttman, Abstr., 1907, ii, 862) obtained when sodium tetrathionate and water are repeatedly evaporated to dryness approximate to the quantities required by the preceding equations. Experiments, in which the prevention of the secondary reaction is attempted by the addition of ammonium hydroxide or sodium hydrogen carbonate, do not give satisfactory results. In the decomposition of sodium tetrathionate by water or alkalis, it seems impossible to stop secondary reactions, but there appears to be no doubt that sulphur is always a product of the primary reaction.

Aromatic disulphides are exceptions to the foregoing rule; by treatment with an alkali, they are decomposed without elimination of sulphur. The author regards the decomposition as occurring initially, thus: $Ph \cdot S \cdot S \cdot Ph + H_2O = Ph \cdot SH + Ph \cdot S \cdot OH$, but the evidence for this view is by no means conclusive. When phenyl disulphide, alcoholic sodium hydroxide, and benzyl chloride are heated in a reflux apparatus, *phenyl benzyl sulphide*, $Ph \cdot S \cdot CH_2Ph$, m. p. 42° , and phenylbenzylsulphone are obtained, without doubt by the action of the benzyl chloride on the initially formed phenyl mercaptan and benzenesulphinic acid respectively (compare Schiller and Otto, this Journ., 1877, 463). Since, however, benzoic acid is also formed from the benzyl chloride

when 4:4'-dinitrodiphenyl disulphide is used in the place of phenyl disulphide (this vol., i, 631), some oxidising agent must be produced in the reaction; this fact furnishes the chief evidence for the author's view quoted above. The hypothetical phenyl derivative of sulphur hydrate, $\text{Ph}\cdot\text{S}\cdot\text{OH}$, being unstable in alkaline solution, changes to phenyl mercaptan and benzenesulphinic acid, somewhat like benzaldehyde in the Cannizzaro reaction. By the decomposition of sodium ethyl thiosulphate by alcoholic sodium hydroxide, Gutmann (Abstr., 1907, i, 671; this vol., i, 497) obtained a solution which oxidised arsenites to arsenates, and probably contained the ethyl derivative, $\text{Et}\cdot\text{S}\cdot\text{OH}$. By treating this solution with benzyl chloride, the author obtains ethyl mercaptan, benzyl ethyl sulphide, benzylethylsulphone, and benzyl disulphide; the formation of these substances is regarded as furnishing additional evidence for the existence of the ethyl derivative of sulphur hydrate. The fission of *p*-tolyl disulphoxide by sodium hydroxide in the presence of benzyl chloride yields *p*-toluenesulphinic acid, *p*-tolyl disulphide, and *p*-tolylbenzylsulphone, the formation of which is due to the action of the benzyl chloride on the sulphinic acid (compare Otto and Rossing, Abstr., 1886, 711; Remsen and Turner, Abstr., 1901, i, 270; Fromm and Palma, Abstr., 1906, i, 819).

[With O. GAUFF.]—Derivatives of sulphonylic acid, H_2SO_2 , have been obtained by several investigators (compare Bazlen, Abstr., 1905, ii, 240; Reinking, Dehnelt, and Labhardt, *ibid.*, i, 261). By the interaction of zinc dust and sulphuryl chloride in dry ether, Fromm and Palma (Abstr., 1906, i, 819) obtained a solution of zinc sulphonylate, which yielded dibenzylsulphone by heating with benzyl chloride and 10% sodium hydroxide. A more searching examination of the reaction has shown the presence of a small amount of benzylsulphinic acid, so that the formation of zinc hyposulphite in the first reaction is not excluded, as previously mentioned. Since, however, sodium or zinc hyposulphite is insoluble in dry ether, or in ether containing water, alcohol, or zinc chloride, the formation of the sulphonylic acid cannot be due to a hyposulphite; its production is explained thus: (I) $\text{Zn} + \text{SO}_2\text{Cl}_2 = \text{ZnCl}_2 + \text{SO}_2$, (II) $\text{SO}_2 + 2\text{NaOH} + \text{CH}_2\text{PhCl} = \text{NaCl} + \text{CH}_2\text{Ph}\cdot\text{SO}_2\text{Na} + \text{H}_2\text{O}$. Magnesium, copper, iron, or sodium have no action on sulphuryl chloride in dry ether, but sodium amalgam reacts vigorously, the product yielding with benzyl chloride and sodium hydroxide, benzylsulphinic acid, the formation of which is again attributed to the liberation of sulphur dioxide.

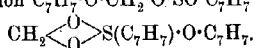
Aldehydes or ketones react with sodium hyposulphite in the presence of sodium hydroxide in accordance with the equation: $\text{RR}'\text{CO} + \text{Na}_2\text{S}_2\text{O}_4 + \text{NaOH} = \text{RR}'\text{C}(\text{OH})\cdot\text{SO}_2\text{Na} + \text{Na}_2\text{SO}_3$ (compare Meister, Lucius & Brüning, Eng. Pat., 1903, 5867; *Chem. Zentr.*, 1906, i, 423; Bazlen, *loc. cit.*). The substance, $\text{RR}'\text{C}(\text{OH})\cdot\text{SO}_2\text{Na}$, may be a hydroxysulphinate, an ester of sodium hydrogen sulphonylate, or a derivative of orthosulphonylic acid (compare Bazlen, *loc. cit.*). To ascertain which representation is correct, the behaviour of Rongalite C (sodium formaldehydesulphonylate), $\text{OH}\cdot\text{CH}_2\cdot\text{SO}_2\text{Na}$, has been studied. The reduction of rongalite by tin and hydrochloric acid, or by hydrogen sulphide in the presence of hydrochloric acid, does not give much information, since the production of the substance obtained, trithio-

formaldehyde, is explicable by each formula. Oxidation by potassium permanganate, or by chlorine or bromine water, causes complete decomposition. Better results are obtained by adding an alcoholic solution of benzyl chloride to rongalite and aqueous sodium hydroxide, and heating for three hours, whereby dibenzylsulphone, sodium benzylsulphonate in small amount, and *dibenzylrongalite*, $C_{15}H_{14}O_3S$, m. p. 80–81°, are obtained; in the absence of the alkali, only the sulphone is formed. The formation of the sulphone in the presence or the absence of sodium hydroxide is evidence for the sulphonylate formula of rongalite: (I) $OH \cdot CH_2 \cdot O \cdot SO \cdot Na + NaOH + 2C_6H_5Cl = SO_2(C_6H_5)_2 + 2NaCl + CH_2O + H_2O$. (II) $OH \cdot CH_2 \cdot O \cdot SO \cdot Na + 2C_6H_5Cl = SO_2(C_6H_5)_2 + NaCl + HCl + CH_2O$.

Dibenzylrongalite is not formed in a secondary reaction between dibenzylsulphone, sodium hydroxide, and rongalite, neither does it yield the sulphone by heating with sodium hydroxide in the presence or the absence of benzyl chloride. Its formation is not due to a reaction between dibenzylsulphone and formaldehyde, for these two substances, heated with 10% sodium hydroxide, form *diformaldibenzylsulphone*, $C_{16}H_{16}O_3S$, m. p. 188°, which from its behaviour receives the constitution $O \langle \begin{smallmatrix} CH_2 \cdot CHPh \\ CH_2 \cdot CHPh \end{smallmatrix} \rangle SO_2$, and is not obtained from

dibenzylrongalite. Treated with hydrogen chloride in glacial acetic acid, dibenzylrongalite yields benzyl chloride (hence one benzyl group is attached to oxygen) and *formaldibenzylsulphoxide*, $C_{15}H_{14}O_3S$, m. p. 108°; the latter is also obtained from dibenzyl disulphoxide, $C_{14}H_{14}O_2S_2$, m. p. 108°, formaldehyde, and hydrogen chloride. Dibenzylrongalite is decomposed by bromine in chloroform in sunlight, yielding benzyl bromide, *benzylsulphonyl bromide*, $C_7H_7 \cdot SO_2Br$, m. p. 79°, and *p*-bromobenzylsulphonic acid. Reviewing the preceding decompositions, the author concludes that rongalite is a sulphonylate,

$OH \cdot CH_2 \cdot O \cdot SO \cdot Na$ or $CH_2 \langle \begin{smallmatrix} O \\ O \end{smallmatrix} \rangle SNa \cdot OH$, and that dibenzylrongalite has the constitution $C_6H_5 \cdot O \cdot CH_2 \cdot O \cdot SO \cdot C_6H_5$ or



Sodium acetonesulphonylate is more stable than rongalite, for a substance analogous to dibenzylrongalite is not obtained when its solution is heated with benzyl chloride and sodium hydroxide. Since, also, sodium benzaldehydesulphonylate (Bazlen, *loc. cit.*) is unchanged when heated at 120° with benzyl chloride and aqueous sodium hydroxide, the author ascribes to these two sulphonylates the formulae of hydroxysulphinates: $OH \cdot CMe_2 \cdot SO_2Na$ and $OH \cdot CHPh \cdot SO_2Na$.
C. S.

Preparation of Sulphinic Acids. EMIL KNOEVENAGEL and JAMES KENNER (*Ber.*, 1908, 41, 3315–3322).—The authors find that aromatic sulphinic acids can be readily prepared in a yield of about 80% by the action (first studied by Friedel and Crafts, *Abstr.*, 1890, 241) of sulphur dioxide and aluminium chloride on aromatic hydrocarbons or their halogen derivatives at a low temperature. The reaction is started by passing dry hydrogen chloride through the

mixture, and the compound of aluminium chloride and aromatic sulphinic acid is decomposed by alkalis. Phenol ethers also yield sulphinic acids very readily, and the use of hydrogen chloride is not necessary, but sulfoxides and sulphonium compounds are also produced. The reaction is probably accompanied by the intermediate formation of the compound $\text{AlCl}_3 \cdot \text{SO}_2\text{Cl}$.

Sulphinic acids were prepared from benzene, *p*-toluene, *o*-, *m*-, and *p*-xylene, mesitylene (m. p. 100° . Holtmeyer, *Zeitsch. Chem.*, 1867, 686, gives $98-99^\circ$), ψ -cumene, *p*-cymene, naphthalene (*a*-acid), *p*-chlorobenzene (m. p. $98-99^\circ$. Oberländer, *Diss.*, Heidelberg, gives 5° lower), and *p*-bromobenzene.

Anisole yields anisolesulphinic acid (m. p. 73° . Kreuder, *Diss.*, Heidelberg, gives $97-98^\circ$) together with di-*p*-anisyl sulfoxide and trianisylsulphonium chloride (isolated as platinichloride). From phenetole a mixture of the sulfoxide and sulphonium compound was obtained. J. C. C.

Sulphinic Anhydrides. EMIL KNOEVENAGEL and LEO POLACK (*Ber.*, 1908, 41, 3323-3331).—When aromatic sulphinic acids are treated in the cold with acetic anhydride and a drop of concentrated sulphuric acid, or a few drops of a 0.1% solution of ferric chloride in acetic acid or acetic anhydride, the corresponding sulphinic anhydrides are produced.

Benzenesulphinic anhydride, $\text{O}(\text{SO} \cdot \text{C}_6\text{H}_5)_2$ (probably previously obtained by Otto and Ostrop, *Annalen*, 1866, 141, 374, as an oil; compare also Otto and Gruber, *ibid.*, 1868, 145, 11), is a white, crystalline substance, m. p. $66-67^\circ$. When preserved in a closed vessel or over concentrated sulphuric acid in a vacuum desiccator, it decomposes with the formation of benzene disulphoxide, $\text{C}_6\text{H}_5 \cdot \text{SO}_2 \cdot \text{S} \cdot \text{C}_6\text{H}_5$, and benzenesulphonic acid, together with compounds of high m. p. The latter are not formed when the substance is kept in a vacuum desiccator over soda-lime, and the above decomposition proceeds more slowly.

Toluene-p-sulphinic anhydride, $\text{C}_6\text{H}_4\text{Me} \cdot \text{SO} \cdot \text{O} \cdot \text{SO} \cdot \text{C}_6\text{H}_4\text{Me}$, has m. p. 75° ; it decomposes after a time into *p*-toluene disulphoxide and a mixture of toluene-sulphinic and -sulphonic acids.

p-Xylenesulphinic anhydride, $\text{O}(\text{SO} \cdot \text{C}_6\text{H}_4\text{Me}_2)_2$, has m. p. $68-69^\circ$. In a vacuum desiccator it decomposes into *p*-xylenesulphonic acid and *p*-xylene disulphoxide, white tablets, m. p. $70-72^\circ$. The same products are formed when *p*-xylenesulphonic acid is heated in a sealed tube at $120-130^\circ$.

ψ -Cumenesulphinic anhydride, $\text{O}(\text{SO} \cdot \text{C}_6\text{H}_3\text{Me}_3)_2$, has m. p. $92-93^\circ$. Mesitylenesulphinic anhydride has m. p. $118-121^\circ$. *p*-Cymenesulphinic anhydride was obtained as an oil, which was probably a mixture of isomerides. *p*-Bromobenzenesulphinic anhydride begins to melt at $79-81^\circ$, and is completely molten at $108-109^\circ$. Even after ten minutes the m. p. is raised to 140° , and after a longer time the substance is converted into *p*-bromobenzene disulphoxide, m. p. 155.5° , which is also formed on heating *p*-bromobenzenesulphinic acid in a sealed tube at $120-130^\circ$. *p*-Iodobenzenesulphinic anhydride, when freshly prepared, begins to melt at $75-80^\circ$, and is completely molten

above 100°. After half an hour the m. p. is 160° (decomp.), and iodobenzene disulphoxide is probably present.

When benzene- or *p*-toluene-sulphinic anhydride is mixed with ammonium carbonate, the ammonium salt of the corresponding sulphonic acid is formed, together with the corresponding disulphoxide. The latter anhydride gives with dry ammonia, ammonium toluene-*p*-sulphonate, together with the corresponding disulphoxide and a trace of the sulphinic acid. When the experiment is carried on after the vessel containing the toluene-*p*-sulphinic anhydride has been filled with dry hydrogen, a small amount of *p*-toluenesulphonamide is formed. Benzenesulphonic anhydride gives with diethylamine the diethylamine salt of benzenesulphonic acid; with aniline, di-*p*-aminophenyl sulphoxide; with *p*-toluidine, the corresponding salt of benzenesulphonic acid, and with phenol, hydroxydiphenyl sulphide.

J. C. C.

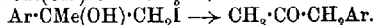
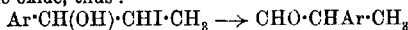
Action of Arsenites and Cyanides on Thiosulphonates. AUGUST GUTMANN (*Ber.*, 1908, 41, 3351—3356).—Sodium toluene-*p*-thiosulphonate is reduced by potassium cyanide or sodium arsenite in sodium hydroxide solution to the *p*-sulphinate, with attendant formation of thiocyanate or thioarsenate at 100°: $C_6H_4Me \cdot S_2O_2Na + Na_3AsO_3 = C_6H_4Me \cdot SO_2Na + Na_3AsO_3S$.

A solution of the toluene-*p*-thiosulphonate, boiled with 10 mols. of sodium hydroxide in 15% solution, becomes lemon-yellow, and on evaporation deposits sulphinate. The yellow solution gives with hydrochloric acid, sulphur and sulphinate, but no hydrogen sulphide; with an alkaline zinc solution, no zinc sulphide, and with arsenite or cyanide, decolorisation at once results. The conclusion is drawn that the yellow colour is due to NaOSH, the *sodium hydropersulphide* reacting thus with cyanide: $NaOSH + KCN = KCNS + NaOH$.

Sodium sulphide also gives a deep yellow solution, which is likewise decolorised by cyanide or arsenite, and is held to contain NaSSH.

W. R.

Transposition of Phenyl. Migration of the Naphthyl Group in Iodohydrins of the Naphthalene Series. MARC TIFFENEAU and DAUDEL (*Compt. rend.*, 1908, 147, 678—680).—The authors find that, exactly as in the cases previously observed with the phenyl group (compare this vol., i, 165, 166), the naphthyl nucleus in derivatives containing the ethylene linking migrates under the influence of iodine and mercuric oxide, thus:



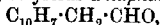
* *a-Allylnaphthalene*, $C_{10}H_7 \cdot CH_2 \cdot CH : CH_2$, prepared by the interaction of allyl bromide and magnesium *a*-naphthyl bromide, has b. p. 265—267°; with mercuric oxide and iodine in aqueous ethereal solution, the *iodohydrin*, $C_{10}H_7 \cdot CH_2 \cdot CH(OH) \cdot CH_2I$, is obtained, which loses its iodine when treated with silver nitrate, but the aldehydic compound is not formed.

a-Naphthylpropylene, $C_{10}H_7 \cdot CH : CH \cdot CH_3$, produced by boiling *a*-allylnaphthalene with alcoholic potassium hydroxide, has b. p. 147—149°/15 mm., and 275—278° under the ordinary pressure, accompanied by

partial polymerisation. When treated with mercuric oxide and iodine, it is directly transformed into α -1-naphthylpropaldehyde, b. p. 170—171°/14 mm., D_4^{20} 1.118 (Darzens, this vol., i, 91), which, on oxidation, yields α -1-naphthylpropionic acid, m. p. 145°.

β -1-Naphthylpropylene, $C_{10}H_7 \cdot CMe \cdot CH_2$, with iodine and mercuric oxide, gives α -naphthylacetone, $C_{10}H_7 \cdot CH_2 \cdot CO \cdot CH_3$, the semicarbazone of which has m. p. 205°.

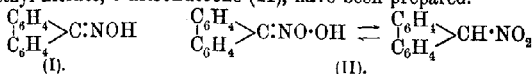
α -Naphthylethylene, $C_{10}H_7 \cdot CH \cdot CH_2$, prepared by the interaction of acetaldehyde and magnesium α -naphthyl bromide, is the chief constituent of the fraction of the products, b. p. 135—138°/15 mm.; with iodine and mercuric oxide, it yields α -naphthylacetaldehyde,



b. p. 163—166°/13 mm. The semicarbazone has m. p. 208°. On oxidation, the aldehyde yields α -naphthylacetic acid. J. C. C.

Condensation of Fluorene with Alkyl Nitrites and Nitrates by means of Potassium Ethoxide. WILHELM WISLICENUS and MARTIN WALDMÜLLER (*Ber.*, 1908, 41, 3334—3340).

—Although fluorene does not react with amyl nitrite (Thiele and Henle, *Abstr.*, 1906, i, 571) or ethyl nitrate (Wieland, *Diss.*, Munich, 1901) in presence of sodium ethoxide, the authors find that the condensation proceeds smoothly when potassium ethoxide is used. From fluorene and amyl nitrite, fluorenoneoxime (I), and from fluorene and ethyl nitrate, 9-nitrofluorene (II), have been prepared.



The two desmotropic forms of the latter have been obtained; the *aci*-form is comparatively very stable. For the condensation, an alcoholic-etheral solution of potassium ethoxide is used.

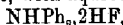
The potassium compound of fluorenoneoxime, $C(C_6H_5)_2 \cdot NOK$, is stable in dry air, and decomposes between 210° and 220°. With water it yields fluorenoneoxime.

The potassium compound of 9-isonitrofluorene (9-*aci*-nitrofluorene) results from the condensation of fluorene and ethyl nitrate in presence of potassium ethoxide. It forms small, yellow needles, and a dilute solution of it gives a dark green coloration with ferric chloride. The corresponding ammonium salt, small, yellow crystals, decomposing at 146—148°, sodium salt, glistening, yellow leaflets, and silver salt are described. 9-isoNitrofluorene (9-*aci*-nitrofluorene), prepared from the sodium salt, forms small, greenish-yellow needles, m. p. 132—135°; it dissolves in potassium hydroxide. 9-Nitrofluorene, formed when an alcoholic solution of the *aci*-compound is kept or is warmed for a short time, crystallises in small, colourless, glistening tablets, m. p. 181—182° (decomp.). It is insoluble in potassium hydroxide, but is dissolved by sodium or potassium ethoxide, regenerating the *aci*-form. An ethereal solution of the *aci*-form gives with dry ammonia an immediate precipitate of the ammonium salt, but the true nitro-compound does not react with ammonia under the same conditions. Phenylcarbimide is without action on the nitro-derivative, whilst with the *aci*-form a vigorous reaction ensues,

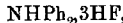
accompanied by evolution of gas. Both forms of nitrofluorene yield fluorenone when carefully heated. When a solution of the potassium compound of the *aci*-form is exposed to the air, or if air is passed through the solution, fluorenone and potassium nitrite are formed; the reaction proceeds more quickly with an absolute alcoholic solution. 9-Bromo-9-nitrofluorene, $\text{C}_6\text{H}_4 > \text{CBr} \cdot \text{NO}_2$, prepared by adding bromine water to an aqueous solution of potassium *aci*-nitrofluorene, forms small, yellow, glistening needles, m. p. 107—108° (decomp.). On heating, bromine and nitrogen dioxide are evolved and fluorenone is formed.

J. C. C.

Hydrofluorides of Some, in Part very Weak, Organic Bases. RUDOLF F. WEINLAND and F. REISCHLE (*Ber.*, 1908, 41, 3671—3674. Compare Weinland and Lewkowitz, *Abstr.*, 1905, i, 518).—The following hydrofluorides have been prepared: From triphenylamine (with concentrated alcoholic hydrofluoric acid), $\text{NPh}_3 \cdot \text{HF}$, bright green powder; from diphenylamine, with aqueous hydrofluoric acid,



small, colourless crystals; with alcoholic hydrofluoric acid,



colourless leaflets; from azobenzene, $\text{N}_2\text{Ph}_2 \cdot \text{HF}$, long, orange-red, glistening needles; from dimethylpyrone, with aqueous hydrofluoric acid, $\text{C}_7\text{H}_8\text{O}_2 \cdot 3\text{HF} \cdot \frac{1}{2}\text{H}_2\text{O}$, large, colourless prisms, and with alcoholic hydrofluoric acid, $\text{C}_7\text{H}_8\text{O}_2 \cdot 3\frac{1}{2}\text{HF} \cdot \frac{1}{2}\text{H}_2\text{O}$, leaflets.

J. C. C.

Studies in Nitration. V. Melting Points of Mixtures of *o*- and *p*-Nitroanilines. J. BISHOP TINGLE and H. F. ROLKER (*J. Amer. Chem. Soc.*, 1908, 30, 1764—1767. Compare *Abstr.*, 1907, i, 120; this vol., i, 408, 778, 893).—In an earlier paper (this vol., i, 408), the melting points of mixtures of *o*- and *m*-, and of *m*- and *p*-nitroanilines were recorded, and it was stated that the curve for mixtures of the ortho- and para-compounds was very irregular. The experiments on *o*- and *p*-nitroaniline have now been repeated, in which the difference in composition between consecutive mixtures was 2%. Curves have been constructed showing the melting points of the original mixtures and the melting points after the fused mixtures had been cooled quickly and then re-melted. It is suggested that the variations in these two series of melting points are due to polymorphism, a substance on melting changing into another form with a different m. p.

The solubility of the nitroanilines in alcohol has been determined. At 15°, 100 c.c. of 95% alcohol dissolve 15.848 grams of *o*-, 4.960 grams of *m*-, and 4.030 grams of *p*-nitroaniline.

E. G.

***o*-Chloroacetanilide and Some Halogenphenylglycines.** CARL G. SCHWALBE, W. SCHULZ, and HERMANN JOCHHEIM (*Ber.*, 1908, 41, 3790—3796).—It is known that the presence of negative groups makes the displacement of a halogen in a benzene derivative more easily effected. It was therefore anticipated that oxindole would be formed on heating *o*-*o*-dichloroacetanilide with copper powder, and that indigo-tin would result from the fusion of *o*-chlorophenylglycine with sodamide.

The halogen is, however, too firmly held, and only a 0.4% yield of indigotin was obtained, and oxindole could only be detected.

o-*Dichloroacetanilide*, $C_8H_4Cl_2NH\cdot CO\cdot CH_2Cl$, prepared either from chloroacetic acid, *o*-chloroaniline, and phosphorus trichloride (compare Janson, Abstr., 1907, i, 312), or from chloroacetyl chloride and *o*-chloroaniline, crystallises from light petroleum in woolly needles, m. p. 67°.

o-*Chlorophenylglycine*, $C_8H_7O_2NCl$, obtained from *o*-chloroaniline by heating with alcoholic potash, formaldehyde, and potassium cyanide (Abstr., 1904, i, 153), crystallises in white leaflets, m. p. 171°. The amide, $C_8H_9ON_2Cl$, forms white, glistening needles, m. p. 142°; the ethyl ester, $C_{10}H_{12}O_3NCl$, is a colourless oil, b. p. 288–291°; *acetyl-o*-chlorophenylglycine, $C_{10}H_{10}O_3NCl$, forms white crystals, decomp. 210°; its ethyl ester, $C_{12}H_{14}O_3NCl$, is a colourless liquid, b. p. 205°/20 mm. The *m*- and *p*-chlorophenylglycines have m. p.'s 93° and 141° respectively; 2 : 4-dichlorophenylglycine, $C_8H_7O_2NCl_2$, crystallises from water; m. p. 127°. The *o*- and *p*-bromophenylglycines, $C_8H_7O_2NBr$, have m. p.'s 157° and 150°. W. R.

Preparation of Aromatic Glycines. GEORGES IMBERT and CONSORTIUM FÜR ELEKTROCHEMISCHE INDUSTRIE (D.R.-P. 199624).—

Aromatic glycines have hitherto been prepared by the interaction of monohalogenated acetic acids and aromatic amines. It has now been found that the former may be replaced by the dihalogenated vinyl ethers, the latter being supposed to change in the following manner: $C_2HCl_2\cdot OEt + H_2O = HCl + CH_2Cl\cdot CO_2Et$. Accordingly, 1 mol. of the dihalogenated vinyl ether is allowed to react with 3 mols. of the aromatic base in aqueous or dilute alcoholic solution, but the excess of base may be replaced by some acid-fixing agent, such as calcium, magnesium, or barium carbonate. Ethyl dichloro- or dibromo-vinyl ether and aniline give rise to a mixture of ethyl phenylglycinate (90%) and phenylglycinanilide (10%). G. T. M.

Diphenylamine Derivatives. FRITZ ULLMANN and REINER DAHMEN (*Ber.*, 1908, 41, 3744–3755).—While attempting to prepare 4-nitrodiphenylamine-2-sulphonic acid by Fischer's method (Abstr., 1892, 331) with the object of obtaining from it the corresponding sulphone by elimination of water, it was found, on boiling the aqueous solution of the acid with a small quantity of sulphuric acid, that the sulpho-group was removed, with the formation of *p*-nitrodiphenylamine. Further investigation has shown that aminodiphenylamine-2-sulphonic acids behave in a similar manner; it has therefore been found possible by this method to prepare the following derivatives of diphenylamine: *p*-nitro-, *p*-amino-, 4-nitro-4'-amino-, 4-nitro-3'-amino-, and 4-nitro-2'-amino-diphenylamine, 4-nitrophenyl-2'-tolylamine, 4-nitrophenyl-4'-tolylamine, and 4-aminophenyl-4'-tolylamine.

The sodium salts of the *o*-sulphonic acids were obtained by the interaction of the base with sodium 2-chloro-5-nitrobenzenesulphonate; in the case of aniline and *o*-toluidine, the condensation was brought about by heating in the presence of calcium carbonate and glycerol; with *p*-toluidine and the three phenylenediamines, the components were simply heated together in aqueous solution with calcium carbonate.

Sodium 4-nitrophenyl-2'-tolylamine-2-sulphonate, $C_{13}H_{11}O_5N_3SN_2$, crystallises in yellow needles. *4-Nitrophenyl-2'-tolylamine*, $C_{13}H_{12}O_2N_2$, forms glistening, dark yellow leaflets, m. p. 115° , and is reduced to *4-aminophenyl-2'-tolylamine*, $C_{13}H_{14}N_2$, white leaflets, m. p. 58.5° , the sulphate of which forms white needles. The *p*-amino-compound may also be obtained from *4-aminophenyl-2'-tolylamine-2-sulphonic acid*, $C_{13}H_{14}O_4N_2S$, which crystallises in small, colourless needles.

Sodium 4-nitrophenyl-4'-tolylamine-2-sulphonate, $C_{13}H_{11}O_5N_3SN_2$, forms orange-red needles with a blue reflex. *4-Nitro-4'-aminodiphenylamine* has m. p. 207° : Bandrowski gives m. p. 211° (Abstr., 1901, i, 48); the hydrochloride, $C_{12}H_{11}O_2N_3 \cdot HCl$, forms dark steel-blue, glittering needles.

4-Nitro-4'-aminodiphenylamine-2-sulphonic acid, $C_{13}H_{11}O_5N_3S$, crystallises in yellowish-brown needles.

4-Nitrophenyl-4'-tolylamine, $C_{13}H_{12}O_2N_2$, crystallises in glistening, yellow needles, m. p. 136° . *4-Aminophenyl-4'-tolylamine-2-sulphonic acid*, $C_{13}H_{14}O_4N_2S$, forms small, white needles; the sodium salt crystallises in colourless leaflets.

4-Nitro-3'-aminodiphenylamine-2-sulphonic acid, $C_{12}H_{11}O_4N_3S$, forms brownish-yellow needles. *4-Nitro-3'-aminodiphenylamine*, $C_{12}H_{11}O_2N_3$, crystallises in glistening, brownish-yellow leaflets, m. p. 156° ; the hydrochloride forms large, deep yellow needles with a blue reflex.

4-Nitro-2'-aminodiphenylamine-2-sulphonic acid, $C_{13}H_{11}O_5N_3S$, crystallises in yellow needles. It is converted when boiled with dilute sulphuric acid into *4-nitro-2'-aminodiphenylamine* (14%) and *4-nitrophenol-2-sulphonic acid*.
W. H. G.

Phenyl-, Naphthyl-, and Menthyl-carbimides. C. VALLÉE (Ann. Chim. Phys., 1908, [viii], 15, 331—432).—The author has made an exhaustive examination of the reactions between certain aryl-carbimides and (1) organic acids (compare Abstr., 1905, i, 771), (2) phenol and some of its substituted derivatives, (3) esters of hydroxy-acids, (4) amino-acids, and of the condensation of *s*-diphenyl-carbamide with certain acyl tartaric anhydrides. The following compounds are described for the first time: *aniline benzenesulphinate*, $PhSO_2H \cdot NH_2Ph$, m. p. 132° ; *aniline hydroxybenzylphosphinite*,
 $OH \cdot CHPh \cdot PHO \cdot OH \cdot NH_2Ph$,

m. p. 99° ; *aniline dihydroxydibenzylphosphinate*,
 $PO(CHPh \cdot OH)_2 \cdot OH \cdot NH_2Ph$,

m. p. 190° ; the diphenylurethane of ethyl dihydroxydibenzylphosphinate, $C_{30}H_{29}O_6N_2P$, m. p. 203° ; phenylurethane of tribromophenol,
 $C_6H_2Br_3 \cdot O \cdot CO \cdot NHPh$,

m. p. 168° ; menthylurethane of trimethylcarbinol,
 $CMe_3 \cdot O \cdot CO \cdot NH \cdot C_{10}H_{19}$,

m. p. 112° , $[\alpha]_D - 55.33^\circ$ in toluene; the menthylurethane derivative of ethyl lactate yields the lactam,
 $CHMe \cdot CHO \cdot CO \cdot N \cdot C_{10}H_{19}$, m. p. 77.5° .

The phenylurethanes of substituted phenols, or of the esters of hydroxy-acids, are readily prepared by heating phenylcarbimide with

the hydroxy-compound in the presence of a small quantity of sodium; the phenylurethane of methyl tartrate crystallises in the monoclinic system [$\alpha : b : c = 1.0544 : 1 : 0.4566$; $\beta = 103^\circ$].

The influence of (a) the solvent, (b) the molecular weight and the position of the substituting radicles, and (c) the ethylenic linking in the radicles on the specific rotation of the numerous optically active urethanes prepared in the course of the work is fully discussed in the paper.

M. A. W.

Preparation of Compounds of *p*-Aminophenol, *p*-Methylaminophenol, and *p*-Phenylenediamine with Sulphurous Acid. SOCIÉTÉ ANONYME DES PLAQUES ET PAPIERS PHOTOGRAPHIQUES A. LUMIÈRE ET SES FILS (D.R.-P. 198497).—Crystalline additive compounds of sulphurous acid with *p*-aminophenol, *p*-methylaminophenol, and *p*-phenylenediamine may be produced either by passing sulphur dioxide into a hot aqueous solution or suspension of these bases, or by treating them with 40% aqueous sodium hydrogen sulphite. The salts of the bases may be employed for this reaction, providing that a mixture of sulphite and hydrogen sulphite is employed.

The compound, $10\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{OH} \cdot \text{H}_2\text{SO}_3$, from *p*-aminophenol, white leaflets, m. p. 184° , is only sparingly soluble in cold water.

The compound, $6\text{NHMe} \cdot \text{C}_6\text{H}_4 \cdot \text{OH} \cdot \text{H}_2\text{SO}_3$, from *p*-methylaminophenol, forms small, colourless, odourless crystals, decomposing at 87° .

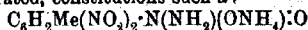
The compound, $9\text{C}_6\text{H}_4(\text{NH}_2)_2 \cdot \text{H}_2\text{SO}_3$, from *p*-phenylenediamine, colourless crystals, m. p. 137° , has a faint odour of sulphur dioxide; it is readily soluble in cold water. These products find employment as photographic developers.

G. T. M.

Abnormal Salts. ANTONI KORCZYŃSKI (*Bull. Acad. Sci. Cracow*, 1908, 633—644).—Finely-powdered phenols and acids have been submitted to the action of dry ammonia. At the ordinary temperature, 1 mol. of ammonia is absorbed by *o*-nitrophenol, 1-nitro-2-naphthol, *o*-nitro-*p*-cresol, dibromo-*o*-nitrophenol, *o*- and *p*-nitrophenolmercurihydroxides, benzoic, cinnamic, *o*- and *m*-nitrobenzoic, 3:5-dinitrobenzoic, *o*- and *p*-chlorobenzoic, and *p*-bromobenzoic acids. *p*-Nitrophenol, 2:4-dinitrophenol, and 3:5-dinitrophenol absorb $1\frac{1}{2}\text{NH}_3$. Two molecules of ammonia are absorbed by *p*-bromo-*o*-nitrophenol, 2:6-dinitrophenol, 3:5-dinitro-*p*-cresol, 5-bromo-3-nitro-*p*-cresol, 4-bromo-2:6-dinitrophenol, picric acid, tribromophenol, trichlorophenol, *p*-nitrobenzoic and 2:4-dinitrobenzoic acids at the ordinary temperature; by *o*-nitro-*p*-cresol, *o*-nitrophenolmercuri-hydroxide, and *p*-nitrophenolmercuri-hydroxide at -10° , and by dibromo-*o*-nitrophenol, *m*- and *p*-nitrobenzoic acids, and *o*-chlorobenzoic acid at -15° . The sodium or potassium salts of the preceding compounds do not absorb ammonia.

Trinitrobenzoic acid absorbs 4NH_3 at 0° , yielding a black substance, which loses 2NH_3 at 25° . Trinitrobenzene and trinitrotoluene at -10 to -15° absorb 2NH_3 , forming reddish-brown, crystalline

substances. For these substances, from which the original compounds cannot be regenerated, constitutions such as



are proposed (compare Hantzsch and Kissel, Abstr., 1900, i, 89).

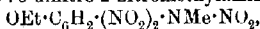
C. S.

New Method of Formation of Ethers of Glycerol with Phenols. PETAR SCHIVKOVITCH (*Monatsh.*, 1908, 29, 951—958).—Brunner obtained a substance, $\text{C}_9\text{H}_{12}\text{O}_4$, during the preparation of quinoldicarboxylic acid by the action of carbon dioxide on quinol in the presence of a hydrogen carbonate and glycerol (Abstr., 1907, i, 319). This substance is probably an ether of glycerol with quinol, but, as it is formed in only very small quantities, it could not be thoroughly investigated. The author has therefore studied the interaction of glycerol with various phenols at 200—210°, and finds that mono-ethers of glycerol are formed.

Phenyl glycerol ether (compare Lindemann, Abstr., 1891, 1198) is formed by heating phenol, glycerol, and sodium acetate together in an atmosphere of hydrogen at 200—210° for twelve to twenty hours. The following derivatives of glycerol are prepared in a similar manner: *o*-tolyl ether, $\text{C}_8\text{H}_9(\text{OH})_2 \cdot \text{O} \cdot \text{C}_6\text{H}_4\text{Me}$, glistening needles, m. p. 66°; *m*-tolyl ether, $\text{C}_{10}\text{H}_{14}\text{O}_3$, white needles, m. p. 65°; *p*-tolyl ether, white needles, m. p. 73—74°; α -naphthyl ether, $\text{C}_{13}\text{H}_{14}\text{O}_3$, white scales, m. p. 91—92°; β -naphthyl ether, $\text{C}_{13}\text{H}_{14}\text{O}_3$, white scales, m. p. 109—110°.

W. H. G.

Preparation of 3-Nitro-2-cyano-1-methoxybenzene, 3-Nitro-2-cyano-1-ethoxybenzene, 4-Nitro-2-cyano-1-methoxybenzene, and 4-Nitro-2-cyano-1-ethoxybenzene. JAN J. BLANKSMA (*Chem. Weekblad*, 1908, 5, 789—795).—The preparation of nitrocyanoderivatives of phenetole and anisole is described. 3-Nitro-2-cyanophenetole (compare Lobry de Bruyn, Abstr., 1885, 656) is prepared from 2:3-dinitrophenetole (this vol., i, 157) by the action of alcoholic ammonia, which yields 2-amino-3-nitrophenetole. The amino-group is replaced by the cyano-group by the Sandmeyer reaction. When boiled with acetic anhydride, 3-nitro-2-aminophenetole yields the corresponding acetyl derivative, which crystallises from light petroleum, m. p. 64°. When heated at 120° in alcoholic solution with methylamine, 2:3-dinitrophenetole yields 3-nitro-2-methylaminophenetole, m. p. 59°, which crystallises from alcohol. Nitric acid, D 1.52, transforms the last compound into 3:5-dinitro-2-nitromethylaminophenetole,

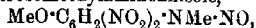


which forms colourless crystals from alcohol, m. p. 79—80° (not 69°, as previously stated, Abstr., 1905, i, 431).

2:3-Dinitroanisole was treated analogously to the corresponding dinitrophenetole with alcoholic ammonia, and yielded 3-nitro-*o*-anisidine, $\text{C}_6\text{H}_4(\text{OMe})(\text{NH}_2)(\text{NO}_2)$, yellow crystals from alcohol or benzene, m. p. 76° (compare Bantlin, Abstr., 1879, 237). The corresponding acetyl derivative was obtained by the action of acetic anhydride; it has m. p. 128°. The acetylation is much facilitated by addition of a drop of concentrated sulphuric acid to the mixture. The acetylation of

3:5-dinitro-*o*-anisidine, $\text{OMe}\cdot\text{C}_6\text{H}_3(\text{NO}_2)_2\cdot\text{NH}_2$, was similarly effected. The compound formed has m. p. 202° .

Exchange of the NH_2 group for CN by Sandmeyer's method converts 3-nitro-*o*-anisidine into 3-nitro-2-cyanoanisole, colourless leaflets, m. p. 171° (de Bruyn, *loc. cit.*). When heated in alcoholic solution with methylamine at 120° , 2:3-dinitroanisole yields 3-nitro-2-methylaminoanisole in the form of dark red needles, readily soluble in alcohol, m. p. 58° . Nitric acid, D 1.52, converts this compound into 3:5-dinitro-2-nitrosomethylaminoanisole,



m. p. 118° .

Reduction of 2:4-dinitrophenetole with ammonium sulphide or sodium disulphide yields 4-nitro-*o*-phenetidine, which by Sandmeyer's reaction is converted into 4-nitro-2-cyanophenetole. It crystallises from boiling water in colourless crystals, m. p. 101° . It is very slightly soluble in cold, but more so in boiling, water, and readily soluble in boiling alcohol. With a mixture of nitric acid and sulphuric acid, it yields 4:6-dinitro-2-cyanophenetole, m. p. 72° (Abstr., 1902, i, 281).

4-Nitro-*o*-anisidine yields analogously 4-nitro-2-cyanoanisole, colourless crystals from boiling water, m. p. 126° , slightly soluble in cold, readily in boiling, alcohol. On treatment with a mixture of nitric acid and sulphuric acid, it is converted into 4:6-dinitro-2-cyanoanisole, which separates from dilute alcohol in colourless crystals, m. p. 71° .

On nitration of 2:3-dinitro-anisole (or -phenetole) and 3-nitro-2-cyano-anisole (or -phenetole), the NO_2 group takes up the para-position with reference to the alkyloxy-group; with 4-nitro-2-cyano-anisole (or -phenetole), it takes up the ortho-position. A. J. W.

Nitration of 5-Nitro-1:3-dimethoxybenzene. JAN J. BLANKSMA (*Rec. trav. chim.*, 1908, 27, 251—256).—When 5-nitro-1:3-dimethoxybenzene (Vermeulen, Abstr., 1906, i, 256) is treated with nitric acid (D 1.52) in the presence of sulphuric acid, 4:5:6-trinitro-1:3-dimethoxybenzene, $\text{C}_6\text{H}(\text{OMe})_2(\text{NO}_2)_3$, m. p. 193° (*Rec. trav. chim.*, 1908, 27, 39), is obtained, together with a small quantity of a compound, m. p. 110° , which has not yet been identified.

The constitution of the trinitro-derivative was determined by converting it by means of alcoholic ammonia into 2:6-dinitro-3:5-dimethoxyaniline, $\text{NH}_2\cdot\text{C}_6\text{H}(\text{OMe})_2(\text{NO}_2)_2$, m. p. 223° ; this yielded 4:6-dinitro-1:3-dimethoxybenzene, m. p. 155° (Abstr., 1902, i, 715), on diazotisation and subsequent treatment with alcohol (this vol., i, 157), and 4:6-dinitroresorcinol, m. p. 210° , was obtained from the latter compound by hydrolysis. The following compounds obtained from 4:5:6-trinitro-1:3-dimethoxybenzene are described:

2:6-Dinitro-3:5-dimethoxymethylaniline, $\text{NHMe}\cdot\text{C}_6\text{H}(\text{OMe})_2(\text{NO}_2)_2$, m. p. 191° ; 2:6-dinitro-1-methylnitroamino-3:5-dimethoxybenzene, $\text{NO}_2\cdot\text{NMe}\cdot\text{C}_6\text{H}(\text{OMe})_2(\text{NO}_2)_2$, m. p. 176° , and 2:6-dinitro-3:5-dimethoxydiphenylamine, $\text{NHPh}\cdot\text{C}_6\text{H}(\text{OMe})_2(\text{NO}_2)_2$, m. p. 190° .

4:5-Dinitro-1:3-dimethoxybenzene, m. p. 131° , is obtained when 5-nitro-1:3-dimethoxybenzene is treated with nitric acid, D 1.45,

and this is converted into the trinitro-compound described above on further nitration with nitric acid (D 1-52).
M. A. W.

Aloesol, a Complex Phenol prepared from Certain Aloes. EUGÈNE LÉGER (*Compt. rend.*, 1908, 147, 806—808. Compare this vol., i, 40).—The compound $C_{11}H_4O_8Cl_4$, obtained previously by the author in the chlorination of Cape or Uganda aloes, is not tetrachloromethoxynaphthaquinone, as the author at first supposed, but a derivative of a new phenol, aloesol. *Tetrachloroaloosol* separates from acetic acid in colourless needles, m. p. 268.9° (corr.). This dissolves in alkalis, giving a yellow solution, from which the original compound is precipitated by the addition of excess of alkali. The *acetyl* derivative, $C_{11}H_3O_8Cl_4Ac$, crystallises in pale yellow prisms, m. p. 125° (corr.).

Dichlorotetrahydroaloosol, $C_{11}H_8O_8Cl_2$, prepared by the action of zinc and acetic acid on tetrachloroaloosol, forms colourless, microscopic needles, m. p. 275° (corr.), and in its behaviour towards alkalis resembles tetrachloroaloosol. The *barium* compound, $(C_{11}H_7O_8Cl_2)_2Ba$, forms long, colourless needles. The *acetyl* derivative occurs in colourless, prismatic needles, m. p. 150 — 151° (corr.). A yellow modification, however, is formed when acetic anhydride is employed, and this furnishes, on hydrolysis, a yellow modification of dichlorotetrahydroaloosol having the same m. p. as the colourless variety.

The presence of a benzene ring in tetrachloroaloosol is indicated by the fact that nitric acid decomposes it with formation of oxalic acid and tetrachloroquinone.
W. O. W.

Preparation of a Compound having the Composition of Nitrosobenzyl Alcohol. KALLE & Co. (D.R.-P. 199317).—The monomeric derivative of *o*-nitrotoluene (compare Abstr., 1907, i, 908) when treated with cold concentrated hydrochloric acid gives rise to an unstable compound, isolated by extraction with ether or distillation in steam, and having the composition of an *o*-nitrosobenzyl alcohol, $NO \cdot C_6H_4 \cdot CH_2 \cdot OH$; this product gives with mercuric chloride an additive compound crystallising from benzene in white needles. When 6% hydrochloric acid is employed in this reaction, the anhydride, anthranil, is obtained.
G. T. M.

***o*, *m*-, and *p*-Tolyethyl Alcohols.** K. KLING (*Bull. Acad. Sci. Cracov.*, 1908, 632—633).—By the energetic electrolytic reduction of *o*-, *m*-, and *p*-tolylacetic acids, the corresponding alcohols have been obtained. *o*-Tolyethyl alcohol, $C_7H_7 \cdot CH_2 \cdot CH_2 \cdot OH$, has b. p. 243 — 243.5° (corr.), D_4^{20} 1.0159, and n_D 1.5214. The *meta*-isomeride has b. p. 242.5 — 243° (corr.), D_4^{20} 1.0127, and n_D 1.5231, and the *para*-compound has b. p. 244.5 — 245° (corr.), D_4^{20} 1.0177, and n_D 1.5271.
C. S.

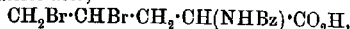
Isolation of Cholesterol from Fats. ERNST SALKOWSKI (*Zeitsch. physiol. Chem.*, 1908, 57, 515—519).—Glikin adversely criticises a method he incorrectly attributes to the author. References are given to an improved method, published in the author's *Festschrift*, and a brief account of it is again published.
W. D. H.

Allylhippuric Acid. A Convenient Material for the Preparation of γ -Disubstituted α -Amino- n -valeric Acids. SÖREN P. L. SÖRENSEN (*Ber.*, 1908, 41, 3387—3390. Compare Abstr., 1905, i, 749; also Fischer and Krämer, this vol., i, 858).—When ethyl allylphthaliminomalonate is hydrolysed with sodium hydroxide and then evaporated with hydrochloric acid, allylglycine is formed, and this, when benzoylated, yields *r*-allylhippuric acid,

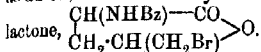


which crystallises from benzene in rectangular plates, m. p. 107—107.5°. The racemic acid can be resolved by means of brucine or quinidine; with the former base, the salt of the *l*-acid crystallises first, and with the latter base, the salt of the *d*-acid.

The active acids combine with bromine, yielding γ -dibromo- α -benzoylaminovaleric acid,



as an oil, and this readily loses hydrogen bromide, forming a crystalline lactone,



It is suggested that this should be used for the syntheses of α -amino- γ -dihydroxy- and other γ -substituted valeric acids. J. J. S.

***o*-Bromophenyl- and α -Bromophenyl-acetamides.** WILHELM STEINKOFF and CZESLAU BENEDEK (*Ber.*, 1908, 41, 3595—3598).—Attempts to prepare nitrated phenylacetamides for the purpose of comparison with nitroacetamide have been unsuccessful. In the course of the work, the authors have prepared two brominated phenylacetamides. These are now described.

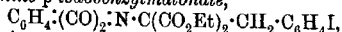
***o*-Bromophenylacetamide**, $\text{C}_6\text{H}_4\text{Br}\cdot\text{CH}_2\cdot\text{CO}\cdot\text{NH}_2$, prepared by the action of bromine on phenylacetamide suspended in water, separates in white crystals, m. p. 181°, and on treatment with sodium nitrite in sulphuric acid solution yields *o*-bromophenylacetic acid.

α -Bromophenylacetamide, $\text{CHBrPh}\cdot\text{CO}\cdot\text{NH}_2$, prepared by treating α -bromophenylacetyl chloride in benzene solution with a current of dry ammonia, forms white crystals, m. p. 143—144°, and on hydrolysis with nitrous acid yields α -bromophenylacetic acid, m. p. 87°.

G. Y.

Halogen Amino-acids: *p*-Iodophenylalanine. HENRY L. WHEELER and SAMUEL H. CLAPP (*Amer. Chem. J.*, 1908, 40, 458—468. Compare this vol., i, 897).—In continuation of their work, the authors have synthesised *p*-iodophenylalanine.

Ethyl phthalimino-*p*-iodobenzylmalonate,



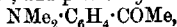
prepared by heating *p*-iodobenzyl bromide with ethyl sodiophthaliminomalonate, forms flat, colourless prisms or six-sided plates, m. p. 112°. On alkaline hydrolysis and subsequent acidification, it gives

phthalamic *p*-iodobenzylacetic acid,
 $\text{CO}_2\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}(\text{CO}_2\text{H})\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\text{I},$
 in needles, m. p. 179°; if, on acidification with hydrochloric acid, the precipitate is digested with the acid for two hours, ***p*-iodophenylalanine**, $\text{CO}_2\text{H}\cdot\text{CH}(\text{NH}_2)\cdot\text{CH}_2\cdot\text{C}_6\text{H}_4\text{I}$, is produced, forming thin scales,

m. p. 270° (decomp.). The *hydrochloride*, thin, flat, colourless plates, decomposing at 248°, and *silver* and *copper* salts are described. The *phenylcarbimide* forms aggregates of flat prisms or plates, m. p. 178—179° (decomp.). The *hydantoin*, $C_{16}H_{12}O_2N_2I$, prepared from this by boiling with 20% hydrochloric acid, forms flat, rhombohedral crystals, m. p. 195—196°. The *ethyl* ester of *p*-iodophenylalanine is a colourless, viscid oil, b. p. 223—226°/25 mm., of which the *picrate* forms flat, yellow plates or tablets, m. p. 200—203°. J. C. C.

p-Dimethylaminocinnamic Acid. LOTTE WEIL (*Monatsh.*, 1908, 29, 895—908).—The object of this investigation was to ascertain if a methylamino- or dimethylamino-group influences the replacement of α -halogen atoms by methoxy- or ethoxy groups in the same manner as alkyloxy-groups (compare Hertzka, *Abstr.*, 1905, i, 291; Werner, *Abstr.*, 1906, i, 180; Goldschmiedt, *Abstr.*, 1907, i, 241). β -Bromo-*p*-dimethylaminocinnamic acid was therefore prepared, and was found to be quite stable towards boiling alcohol and aqueous potassium hydroxide, so that a *p*-dimethylamino-group does not exert the same influence on an α -halogen atom as a *p*-methoxy-group. The *p*-dimethylamino-group was found, however, to have a great influence on the alkylcarboxy-group, for methyl *p*-dimethylaminocinnamate cannot, like methyl cinnamate, be converted by ammonia into the corresponding amide.

p-Dimethylaminocinnamic acid, $NMe_2 \cdot C_6H_4 \cdot CH:CH \cdot CO_2H$, may be prepared by Perkin's method provided potassium acetate is used in the place of the sodium salt; it crystallises in glistening, yellow leaflets, m. p. 216° (decomp.); the *silver* salt is yellow. The *ethyl* ester, $C_{13}H_{17}O_2N$, is most readily obtained by the action of sodium on a mixture of ethyl acetate and *p*-dimethylaminobenzaldehyde; it crystallises in golden-yellow leaflets, m. p. 74—75°, b. p. 205—208°/19 mm.; the *picrate*, $C_{13}H_{17}O_2N \cdot C_6H_5O_7N_3$, forms red crystals, m. p. 130—132°. The *methyl* ester, $C_{12}H_{15}O_2N$, forms pale yellow crystals, m. p. 134—136°; it is converted by bromine in chloroform into a bromo-derivative, which forms colourless crystals, m. p. 164—168° (decomp.), and is either *methyl* $\alpha\beta$ -dibromo-*p*-dimethylamino- β -phenylpropionate, $NMe_2 \cdot C_6H_4 \cdot CHBr \cdot CHBr \cdot CO_2Me$, or *methyl* β -bromo-*p*-dimethylaminocinnamate hydrobromide, $NMe_2 \cdot C_6H_4 \cdot CBr:CH \cdot CO_2Me \cdot HBr$. The substance just described is converted by boiling water, alcohol, or aqueous potassium hydroxide into *methyl* β -bromo-*p*-dimethylaminocinnamate, $NMe_2 \cdot C_6H_4 \cdot CBr:CH \cdot CO_2Me$, an exceedingly stable compound crystallising in glistening, yellow leaflets, m. p. 96°; it is not affected by aqueous potassium hydroxide, but is converted by alcoholic potassium hydroxide into β -bromo-*p*-dimethylaminocinnamic acid, $C_{11}H_{12}O_2NBr$, yellow crystals, m. p. 165°, and *p*-dimethylaminoacetophenone,



crystallising in long needles, m. p. 103°. The latter compound may also be obtained by treating *p*-aminoacetophenone with methyl sulphate; the *phenylhydrazone* forms pale yellow crystals, m. p. about 150°. The substance, m. p. 59°, described by Klingel (*Abstr.*, 1886, 61) is not *p*-dimethylaminoacetophenone, as stated by this author.

p-Dimethylamino- β -phenylpropionic acid, $NMe_2 \cdot C_6H_4 \cdot C_2H_4 \cdot CO_2H$, is obtained as its sodium salt by treating ethyl *p*-dimethylamino-

cinnamate in alcohol with sodium; it crystallises in colourless leaflets, m. p. 104°.

p-Dimethylaminobenzaldehyde-*p*-bromophenylhydrazone, $C_{16}H_{18}N_2Br$, crystallises in brown, felted needles, m. p. 181°.

p-Aminoacetophenonephenylhydrazone has m. p. 112°. W. H. G.

Action of Nascent Hypiodous Acid on Unsaturated Acids.

J. BOUGAULT (*Ann. Chim. Phys.*, 1908, [viii], 15, 296—312).—A detailed account of work already published (compare this vol., i, 179, 269, 340, 791). The action of iodine in the presence of sodium carbonate or hydrogen carbonate on certain β -unsaturated acids is quantitative, and can be employed for the volumetric estimation of hydropiperic and phenylisocrotonic acids.

M. A. W.

Preparation of Arylthioglycollic [Arylthiolacetic] Acids.

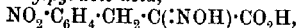
KALLE & Co. (D.R.-P. 201231 and 201232. Compare this vol., i, 672).—The condensation of a diazonium salt with a thioglycollic acid occurs more readily and at lower temperatures (20—25°) in the presence of "molecular copper." A good yield of *o*-carboxyphenylthiolacetic acid is obtained by condensing diazotised anthranilic acid with thioglycollic acid in warm alkaline solution; the intermediate product is precipitated by acids, dissolved in aqueous sodium carbonate, and the solution heated to boiling until all the nitrogen is expelled, when the final product is obtained by cooling and acidifying.

G. T. M.

Some Oximes of the *o*-Nitrotoluene Series and their Changes. ARNOLD REISSERT (*Ber.*, 1908, 41, 3810—3816).—On boiling with water, the oxime of *o*-nitrophenylglyoxylic acid yields *o*-nitrobenzonitrile and carbon dioxide, and this oxime was supposed to be formed as an intermediate product in the conversion of *o*-nitrophenylpyruvic acid oxime by nitrous acid into the *o*-nitrobenzonitrile (Abstr., 1897, i, 417). Further inquiry has, however, shown that by slowly adding a sodium nitrite solution at the bottom of a dilute hydrochloric acid solution of the *o*-nitrophenylpyruvic acid at 60° and afterwards heating to 100°, a 90% yield of *o*-nitrobenzonitrile and oxalic acid is obtained. The oxalic acid can only be produced by the decomposition of the oximino-*o*-nitrobenzoylglyoxylic acid, thus:

$NO_2 \cdot C_6H_4 \cdot C(\cdot NOH) \cdot CO \cdot CO_2H = NO_2 \cdot C_6H_4 \cdot CN + C_2O_2H_2$,
and the assumption of the intermediate formation of the nitrophenylglyoxylic acid is incorrect.

Oximino-*o*-nitrophenylpyruvic acid,



obtained from the acid and hydroxylamine, crystallises in almost colourless needles, m. p. 161° (decomp.), and is hydrolysed by mineral acids into its components. When, boiled with water, it is slowly converted into *o*-nitrophenylacetoneitrile. A small quantity of *o*-nitrophenylacetamide, $C_8H_8O_3N_2$, colourless crystals, m. p. 160—161°, was also isolated; it was also obtained by heating ethyl *o*-nitrophenylacetate with alcoholic ammonia under pressure at 100°. The nitrophenylacetoneitrile is formed by heating the oxime of nitrophenylpyruvic acid at 140—145°.

* The liability of these oximes to change into nitriles depends on the presence of negative contiguous groups, CO_2H and $\text{CO}\cdot\text{CO}_2\text{H}$, as *o*-nitrobenzaloxime is more stable. The conversion of this oxime into *o*-nitrobenzonitrile or its amide is accomplished if small quantities of alkaline substances, like borax, potassium cyanide, or sodium carbonate, are added to the water and the mixture heated for some hours.

W. R.

Preparation of *o*-Carboxyphenylthioglycolic [*o*-Carboxy-phenylthiolacetic] Acid. KALLE & Co. (D.R.-P. 199249).—*o*-Carboxyphenylthiolacetic acid, $\text{CO}_2\text{H}\cdot\text{C}_6\text{H}_4\cdot\text{S}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, yellow crystals, m. p. 213° , was obtained by the interaction of the sodium salts of *o*-thiolbenzoic and chloroacetic acids in aqueous solution; it finds employment in the production of dyes and pharmaceutical products.

G. T. M.

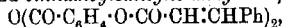
Preparation of the Anhydrides of Acylsalicylic Acids. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. (D.R.-P. 201325 and 201326).—The therapeutic employment of the acylsalicylic acids is attended with two disadvantages—their distinctly acid taste and their appreciably irritating action on the sensitive mucous lining of the stomach. Their anhydrides, which have now been prepared, are free from these disagreeable properties.

Acetylsalicylic [*o*-acetoxybenzoic] **anhydride**, $\text{O}(\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{OAc})_2$, white crystals, m. p. 85° , is obtained by condensing acetylsalicylic acid in benzene solution with thionyl chloride, carbonyl chloride, trichloromethyl formate, or hexachlorodimethyl carbonate in the presence of pyridine.

Acetylsalicylyl [*o*-acetoxybenzoyl] **chloride**, colourless crystals, m. p. $43\text{--}44^\circ$, b. p. $135^\circ/12\text{ mm.}$, from phosphorus pentachloride and acetylsalicylic acid, when warmed with this acid (1 mol.) and dimethylaniline in toluene solution, or when heated with sodium acetylsalicylate, also gives rise to the same anhydride.

The **ethyl carbonate** compound, $\text{O}(\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{O}\cdot\text{CO}_2\text{Et})_2$, clusters of needles, m. p. 74° , was obtained from the corresponding acid by the action of sulphuryl chloride and pyridine in benzene solution.

Benzoylsalicylic anhydride, m. p. $106\text{--}107^\circ$, was prepared from the corresponding acid by means of carbonyl chloride and quinoline in benzene solution, and **cinnamoylsalicylic anhydride**,



m. p. $114\text{--}116^\circ$, was obtained from cinnamoylsalicylic acid (Proc., 1906, 22, 317) by the action of carbonyl chloride and antipyrine in benzene solution.

Other condensing agents, such as acetyl chloride, phosphoryl chloride, and phosphorus tribromide and trichloride, may be employed in the production of these anhydrides.

G. T. M.

Rule in Benzoylation of Aromatic Hydroxy-acids and their Esters. LASSAR-COHN and JOSEF LÖWENSTEIN (Ber., 1908, 41, 3360—3367).—It has been found that the hydroxyl cannot be benzoylated in a hydroxy-compound containing a carboxyl or a

sulphonic group in any position in the ring by the Schotten-Baumann method, but that its esters give benzoates. The following benzoates have been prepared.

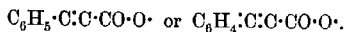
Benzoylsalicylic [*o*-benzoyloxybenzoic] acid, $C_{14}H_{10}O_4$, obtained in small yield by stirring together dipotassium salicylate and benzoyl chloride in light petroleum, crystallises from dilute alcohol; m. p. 132° . It is easily hydrolysed by alkali. The greater portion of the product of the reaction is insoluble in water, and when distilled gives benzoic acid and its phenyl ester. Methyl benzoylsalicylate (benzosalin: D.R.-P. 169247) is obtained in quantitative yield by the Schotten-Baumann method; m. p. 92° , b. p. $350-358^\circ$ (decomp.), $270-280^\circ/120$ mm. *Ethyl p*-benzoyloxybenzoate, $C_{16}H_{14}O_4$, has m. p. 94° , b. p. $358-360^\circ$; the ester of *m*-hydroxybenzoic acid is hydrolysed under the conditions of the experiment, and no benzoyl derivative was obtained.

Ethyl β -benzoyloxynaphthoate, $C_{20}H_{16}O_4$, has m. p. 98° and b. p. $342-345^\circ$. The α -hydroxy- β -naphthoic acid (Abstr., 1888, 60) is considered to be the 2-hydroxynaphthalene-1-carboxylic acid because it is not esterified by hydrogen chloride and alcohol. The *ethyl* ester, $C_{13}H_{12}O_3$, has m. p. 46° , and gives the *benzoate*, $C_{20}H_{16}O_4$, m. p. 51° .

Crude *ethyl phenol-p*-sulphonate, obtained by the action of *ethyl iodide* on the silver salt, gives the *benzoate*, $C_{15}H_{14}O_5S$, m. p. 62° .
W. R.

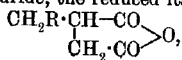
Simultaneous Formation of Isomeric Substitution Products of Benzene. ARNOLD F. HOLLEMAN. XI. Nitration of Phthalic and *iso*Phthalic Acids. J. HUISINGA (*Rec. trav. chim.*, 1908, 27, 260-286).—A detailed account of work already published (Abstr., 1907, i, 136). The percentage solubilities of 3-nitrophthalic, anhydrous 5-nitroisophthalic, hydrated 5-nitroisophthalic, 4-nitroisophthalic, and 2-nitroisophthalic acids at 25° are 2.048, 0.220, 0.157, 0.967, and 0.216 respectively.
M. A. W.

Colour Reactions during the Hydrolysis of Acid Anhydrides. HANS STOBBE (*Ber.*, 1908, 41, 3720-3725).—In the hydrolysis of certain colourless anhydrides to the colourless salts of the corresponding dibasic acids by means of alkali hydroxides, the author has noticed the appearance of an evanescent coloration. The actual colour depends on the particular anhydride, and may be yellow, red, violet, or blue. It is shown that the production of a coloration is characteristic of anhydrides containing either of the following groupings:



Examples of compounds which give such colorations are phenylitaconic anhydride, the two stereoisomeric phenylmethylitaconic anhydrides, diphenylitaconic anhydride, γ -diphenyleneitaconic anhydride, and the anhydrides of 1 phenyl-1 : 2-dihydronaphthalene-2 : 3-dicarboxylic acid and the corresponding 1 : 4-diphenyl-2 : 3-dicarboxylic acid.

Dimethylitaconic anhydride, the reduced itaconic anhydrides,



and the anhydrides of tetrahydronaphthalenedicarboxylic acids do not give colorations.

Experiments have been made by mixing together absolute alcoholic sodium hydroxide with solutions of the anhydrides in various solvents, and determining the time during which the coloration persisted. It was found that in the presence of acetone the colour was retained for a much longer time, especially when the volume of acetone used was relatively large. In all experiments, 1 c.c. of *N*/10 sodium hydroxide and 0.0003 gram of anhydride were used.

In the case of the two stereoisomeric phenylmethylitaconic anhydrides, the colorations were so fugitive that even with acetone the solutions became quite colourless after a few seconds.

Alcohols have a similar effect to ketones, but benzene, chloroform, and carbon tetrachloride have little effect. Alcoholic solutions of other bases have also been used.

J. J. S.

Synthesis of Trimethylhomogallic Acid (Methyliridic Acid). FERDINAND MAUTNER (*Ber.*, 1908, 41, 3662—3665).—3:4:5-Trimethoxybenzaldehyde (this vol., i, 348), hippuric acid, anhydrous sodium acetate, and acetic anhydride, when heated on the water-bath for

one and a-half hours, yield the substance, $C_6H_2(OMe)_3 \cdot CH_2 \cdot C \begin{smallmatrix} \nearrow CO \\ \nwarrow NBr \end{smallmatrix}$, m. p. 165—166°, which is hydrolysed by dilute sodium hydroxide, ammonia, benzoic acid, and 3:4:5-trimethoxyphenylpyruvic acid, $C_6H_2(OMe)_3 \cdot CH_2 \cdot CO \cdot CO_2H$, m. p. 167—168°, being formed. The last-mentioned acid forms an oxime, $C_{12}H_{13}O_8N$, m. p. 155—156°, and with *o*-phenylenediamine hydrochloride, yields a trimethoxybenzylquinazoline, $C_{18}H_{18}O_4N_2$, m. p. 196—197°, and is converted by hydrogen peroxide in alkaline solution into trimethylhomogallic [3:4:5-trimethoxyphenylacetic] acid, $C_6H_2(OMe)_3 \cdot CH_2 \cdot CO_2H$. C. S.

[Preparation of Triphenylmethane Colouring Matters from Diortho-substituted Benzaldehydes]. ANILINFARBEN- & EXTRACT-FABRIKEN VORMALS. J. R. GEIGY IN BASEL (D.R.-P. 199943).—The diortho-substituted benzaldehydes, when condensed with *o*-cresotic acid, give rise to triphenylmethane colouring matters, which have valuable tinctorial properties, being faster to light and alkalis, and dyeing wool in acid-bath in deeper shades, than those from the mono-ortho-substituted benzaldehydes. The following new aldehydes are described:

2:6-Dichlorobenzaldehyde, colourless needles, m. p. 70—71°; 2:6-dichloro-3-nitrobenzaldehyde, colourless leaflets, m. p. 76—77°; 2:6-dichlorobenzaldehyde-3-sulphonic acid; 2-chlorobenzaldehyde-6-sulphonic acid; benzaldehyde-2:6-disulphonic acid, and 6-chlorobenzaldehyde-2:4-disulphonic acid; the sodium salts of all these acids, excepting the last, are salted out in colourless needles; 2:3:6- and 2:4:6-trichlorobenzaldehydes form colourless needles, melting at 86—87° and 58—59° respectively. G. T. M.

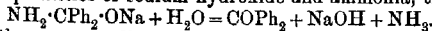
Condensation of cyclopentanone with Benzaldehyde. MAX KAUFFMANN (*Ber.*, 1908, 41, 3726—3727).—In the preparation of dibenzylidenecyclopentanone (Vörländer and Hobohm, *Abstr.*, 1896, i,

603), it is necessary that the alkali hydroxide solution used should not be too dilute. The author finds that very dilute alkali solutions, or solutions of weak alkalis, such as ammonia, trimethylamine, or sodium carbonate, bring about condensations, but that the products formed are quite different from the sparingly soluble dibenzylidenecyclopentanone. Some of these products are oily, others have comparatively low m. p.'s; some are colourless, whereas others are yellow. J. J. S.

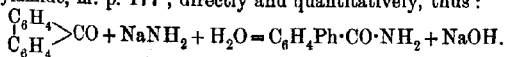
Preparation of Colouring Matters of the Thioindigo-Red Series. FARBERWERKE VORM. MEISTER, LUCIUS & BRÜNING (D.R.P. 198509).—*Bis-o-thioacetophenone*, $S_2(C_6H_4 \cdot COMe)_2$, a substance analogous to indigo-red, is produced by passing air through a solution of *o*-thioacetophenone in aqueous sodium hydroxide. Other oxidising agents, such as alkali ferricyanides, persulphates, perborates, or percarbonates, may be employed. This red colouring matter is also obtained by heating *o*-thioacetophenone with crystallised sodium sulphide; this operation gives rise to the leuco-derivative, which is oxidised as before by aerial oxidation in alkaline solution.

G. T. M.

Products of the Action of Sodamide on Ketones. ALBIN HALLER and ED. BAUER (*Compt. rend.*, 1908, 147, 824–826. Compare Abstr., 1906, i, 441).—When a benzene or toluene solution of benzophenone is heated to boiling with sodamide, in some cases an abundant white precipitate is formed, with or without the previous production of an intense blue coloration, whilst in other cases the sodamide slowly dissolves with or without the subsequent separation of a deposit. The production of the blue colour is probably due to the action of free sodium contained in the sodamide (compare Schorigin, this vol., i, 866). The more nearly anhydrous the solution the slower the precipitate is in forming. By collecting the precipitate, drying, and treating with water, benzamide, mixed with a little benzoic acid, is formed. When decomposed with dilute ammonia, benzamide only is formed. The reactions can be represented thus: $COPh_2 + NaNH_2 = NH_2 \cdot CPh_2 \cdot ONa$; $NH_2 \cdot CPh_2 \cdot ONa + H_2O = NH_2 \cdot CPh_2 \cdot OH + NaOH$. If freshly-prepared sodamide free from sodium is used, and the solvent is absolutely anhydrous, no deposit is formed in the hot solution, but the substance separates after a time as an abundant crystalline crust of the composition $NH_2 \cdot CPh_2 \cdot ONa$, which, when treated with water, regenerates the whole of the benzophenone, and gives theoretical quantities of sodium hydroxide and ammonia, thus:



Under the same conditions, phenyl *p*-tolyl ketone gives an almost equimolecular mixture of benzamide and toluoylamide, whilst phenyl anisyl ketone yields rather more anisamide than benzamide. Fluorenone, when treated in toluene solution with sodamide, gives diphenyl-*o*-carboxylamide, m. p. 177°, directly and quantitatively, thus:



E. H.

Action of Alcoholic Potassium Hydroxide Solution on Ketones.
 P. J. MONTAGNE (*Rev. trav. chim.*, 1908, 27, 327—359. Compare Abstr., 1907, i, 854).—Zagumenny (Abstr., 1877, 459) has shown that benzhydrol is produced when benzophenone is heated at 160° with alcoholic potassium hydroxide. The author finds the same reduction occurs when the ketone is boiled for two days with a 20% alcoholic solution of potassium hydroxide, and that substituted derivatives of benzophenone, when similarly treated, are either reduced similarly or are decomposed, yielding benzoic acid and the substituted benzene. Thus 4-chloro-, 4-bromo-, 4-methyl-, or 4-phenyl-benzophenone yields the corresponding substituted benzhydrol, whilst 2:4:6-trichlorobenzophenone yields trichlorobenzene and benzoic acid; 2:4:6-tribromobenzophenone yields 4-bromobenzhydrol; 2:4:6-trimethylbenzophenone remains unaltered, and phenyl α -naphthyl ketone yields phenyl- α -naphthylcarbinol.

The following new compound was prepared in the course of the work: 2:4:6-tribromobenzophenone, $C_6H_2Br_3 \cdot C(=O)Ph$, obtained by heating benzoyl chloride, 1:3:5-tribromobenzene, and aluminium chloride at 150—160°, forms colourless, shining, triclinic crystals [F. M. JAEGER, $a:b:c = 1.3939:1.11065$; $\alpha = 130^\circ 36'$, $\beta = 122^\circ 59'$, $\gamma = 58^\circ 58'$], $D^{20}_D 2.034$, m. p. 147°, b. p. 405°/765 mm. The same compound was also prepared from 2:4:6-tribromobenzoyl chloride (Sudborough, *Trans.*, 1895, 67, 596) and benzene in the presence of aluminium chloride. The crystallographic measurements by F. M. Jaeger of the following compounds are recorded: 2-bromobenzophenone forms large, colourless, monoclinic prisms [$a:b:c = 0.5045:1.0332$; $\beta = 83^\circ 24.5'$], $D^{20}_D 1.517$; phenylindoxazen forms colourless, rhombic crystals [$a:b:c = 0.7905:1.12600$], $D^{20}_D 1.295$; 2:4:6-tribromobenzonitrile crystallises in the monoclinic system [$a:b:c = 1.2113:1.11025$; $\beta = 44^\circ 23.75'$]; 2:4:6-tribromobenzamide crystallises from alcohol in long needles, which, in contact with the mother liquor, change into monoclinic prisms [$a:b:c = 2.1655:1.11092$; $\beta = 83^\circ 45'$]; 2:4:6-tribromobenzoyl chloride forms colourless, triclinic prisms [$a:b:c = 1.9341:1.10041$; $\alpha = 89^\circ 54.6'$, $\beta = 108^\circ 43.75'$, $\gamma = 84^\circ 21'$].
 M. A. W.

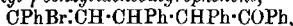
Reactions of Organic Magnesium Compounds with Cinnamylidene Esters. II. Reactions with Methyl α -Phenylcinnamylideneacetate. MARIE REIMER and GRACE POTTER REYNOLDS (*Amer. Chem. J.*, 1908, 40, 428—444. Compare Abstr., 1907, i, 852).—It was shown previously that the compounds produced by the action of organic magnesium compounds on methyl cinnamylidenemalonate are formed by 1:4-addition, and the authors have now extended the reaction to the case of methyl α -phenylcinnamylideneacetate. The reaction proceeds with much greater difficulty than in the previous cases examined, and the resulting compounds are not esters, formed by 1:4-addition, but ketones or tertiary alcohols, formed by replacement of the methoxyl group and subsequent addition of a second molecule of reagent in the 1:4- or 1:2-position, according to the nature of the magnesium compound.

α,β-Diphenyl-γ-benzylidenebutylphenone,
 $\text{CHPh}\cdot\text{CH}\cdot\text{CHPh}\cdot\text{CHPh}\cdot\text{COPh},$

prepared by the action of magnesium phenyl bromide (3 mols.) on methyl *α*-phenylcinnamylideneacetate (1 mol.) in boiling ethereal solution, crystallises in slender, soft, white needles, m. p. 191·5—192·5°. When oxidised with permanganate, it yields *β*-benzoyl-*α,β*-diphenylpropionic acid, $\text{CHPh}\cdot\text{CH}\cdot\text{CHPh}\cdot\text{CO}_2\text{H}$, which crystallises with 1EtOH in small, shining needles, m. p. 211—212°; the methyl ester forms shining, white needles, m. p. 159°.

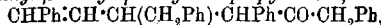
α,β-Diphenyl-γ-benzylidenebutylphenone reacts with magnesium phenyl bromide, yielding a tertiary alcohol which could not be isolated in the pure state; on oxidation, it gives *αβγγ*-tetraphenylbutyrolactone, $\text{CHPh}\cdot\text{CO}\begin{matrix} \diagup \\ \diagdown \end{matrix} \text{O}$, crystallising in slender needles, m. p. 221—221·5°, and, on distillation under diminished pressure, it yields a yellow oil, from which separates a compound, $\text{C}_{22}\text{H}_{18}\text{O}$, probably phenylbenzylidenepropiophenone, crystallising in needles, m. p. 134—135°.

Bromo-α,β-diphenyl-γ-benzylidenebutylphenone,

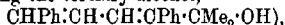


or, less probably, $\text{CHPh}\cdot\text{CBr}\cdot\text{CHPh}\cdot\text{CHPh}\cdot\text{COPh}$, forms white needles, m. p. 186—187°.

Benzyl α-phenyl-β-benzyl-γ-benzylidenepropyl ketone,



prepared by the interaction of magnesium benzyl bromide and methyl *α*-phenylcinnamylideneacetate, forms slender, white needles, m. p. 150°. Magnesium *o*-tolyl or naphthyl bromides do not react with the ester, but magnesium methyl iodide in excess furnishes an oil (probably containing the tertiary alcohol,

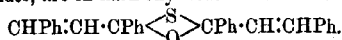


which, on distillation under reduced pressure, loses water and yields a hydrocarbon, $\text{CHPh}\cdot\text{CH}\cdot\text{CH}\cdot\text{CPh}\cdot\text{CMe}\cdot\text{CH}_2$, white crystals, m. p. 97—98°, b. p. 140—150°/25 mm. J. C. C.

Thio-derivatives of Ketones. VI. EMIL FROMM (*Ber.*, 1908, 41, 3644—3661).—Whilst two isomeric forms of trithioaldehydes are known in accordance with stereochemical views, attempts to obtain stereoisomerides of trithioketones, derived from ketones of the type $\text{R}\cdot\text{CO}\cdot\text{R}'$, have hitherto been unsuccessful (compare Fromm and Baumann, *Abstr.*, 1895, i, 362; Fromm and Ziersch, *Abstr.*, 1906, i, 930; Fromm and Höller, *Abstr.*, 1907, i, 710).

[With W. LAMBRECHT].—The authors have now succeeded in obtaining two stereoisomeric, half-sulphurised thioketones by the action of ammonia and hydrogen sulphide on an alcoholic solution of benzylidenacetophenone. The one or the other isomeride is obtained according as the ammonia or the hydrogen sulphide is introduced first into the alcoholic solution. If the solution is saturated with hydrogen sulphide, cooled to 0°, treated with a large excess of ammonia, and left overnight, a quantitative yield of *β*-duplobenzylidenacetophenone sulphide [*monothioduplobenzylidenacetophenone*], $\text{C}_{30}\text{H}_{24}\text{OS}$, m. p. 181°, is obtained as an amorphous, white powder. The *α*-isomeride, m. p. 96°,

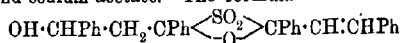
is obtained when an alcoholic solution of benzylidenacetophenone is saturated with ammonia, cooled, and treated with hydrogen sulphide. The two substances show the same reactions. For example, dry distillation causes the formation of benzylidenacetophenone and unexamined products containing sulphur; reduction by zinc dust and glacial acetic acid yields benzylacetophenone, whilst the action of phenylhydrazine in boiling glacial acetic acid results in the formation of hydrogen sulphide and triphenylpyrazoline. These reactions, which show that the skeleton of benzylidenacetophenone is still existent in the two isomerides, are in harmony with the formula



It is a matter of doubt whether the stereoisomerism is connected with the ethylenic linkings or is conditioned by the disposition of the groups about the plane of the heterocyclic ring. Since, however, the more fusible α -duplobenzylidenacetophenone sulphide is transformed into the β -isomeride by the rapid evaporation of its solution in chloroform containing iodine, the latter view is the more favoured, the α -isomeride having the *cis*-configuration.

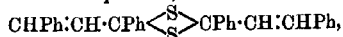
If a chloroform solution of either isomeride is treated with bromine in chloroform, and alcohol is then added, sulphur is eliminated, the solution becomes decolorised, and Rupe and Schneider's β -bromo- α -benzylacetophenone (Abstr., 1895, i, 361) is precipitated. In the absence of the alcohol, decolorisation occurs more slowly, hydrogen bromide is evolved, and a *tribromide*, $\text{C}_{30}\text{H}_{25}\text{OBr}_3\text{S}$, m. p. 147° , is obtained. The α - or the β -isomeride with excess of iodine in chloroform yields a *substance*, $\text{C}_{30}\text{H}_{25}\text{O}_3\text{S}$, m. p. 191° .

When chloroform solutions of the α - and β -isomerides are oxidised by potassium permanganate and dilute sulphuric acid, stereoisomeric oxidation products are obtained. This result is noteworthy, since stereoisomeric trithioaldehydes yield the same oxidation product. The *substance*, $\text{C}_{30}\text{H}_{26}\text{O}_4\text{S}$, m. p. 216° , obtained by the oxidation of β -duplobenzylidenacetophenone sulphide, forms small, white prisms, whilst the oxidation product of the α -isomeride, m. p. 198° , crystallises in needles and is transformed by iodine into its isomeride. Little has been settled regarding the constitution of these oxidation products; they are stable to phenylhydrazine, or benzoyl chloride and sodium hydroxide, or hydrogen chloride in cold glacial acetic acid. They yield benzylidenacetophenone by treatment with hot acetic anhydride and sodium acetate. The formula



is proposed, but is contrary to the fact that the oxidation products cannot be acetylated or benzoylated.

Duplobenzylidenethioacetophenone,



m. p. 191° , is obtained in a bad yield when hydrogen chloride, followed by an excess of hydrogen sulphide, is led into an alcoholic solution of benzylidenacetophenone at 0° ; it is decomposed by

phenylhydrazine, yielding hydrogen sulphide and a substance which is probably triphenylpyrazoline.

[With JAMES L. MCKEE.]—With the hope of preparing another compound possessing the peculiar additive properties of duplobenzylidenethioacetone (Fromm and Höller, *loc. cit.*), the authors have examined the reaction between dibenzylidenacetone and hydrogen sulphide in acid and in ammoniacal solution. Complex compounds are obtained which do not show any marked additive powers, and for which constitutional formulæ are suggested.

Duplodibenzylidenacetoneoxytriimine, $C_{31}H_{36}ON_3$, decomposing at 97° , is an amorphous, yellowish-brown substance obtained by the action of ammonia on an alcoholic suspension of dibenzylidenacetone. It readily loses ammonia when its ethereal solution is treated with hydrogen chloride, whereby the *hydrochloride*, $C_{34}H_{32}ON_3 \cdot 2HCl$, decomp. 185° , is precipitated, from which ammonium hydroxide liberates *duplodibenzylidenacetoneoxydiamine*, $C_{34}H_{32}ON_2$, which decomposes at 137° . When an alcoholic suspension of dibenzylidenacetone is treated with ammonia, cooled, and then treated with hydrogen sulphide, a yellowish-white precipitate of *duplodibenzylidenethioacetonediamine*, $C_{34}H_{34}N_2S_2$, is obtained, which readily loses hydrogen sulphide and takes up water, yielding *duplodibenzylidenethioacetonediamine*, $C_{34}H_{34}ON_2S$, which decomposes at $103-119^\circ$, and is best purified through the *hydrochloride*, $C_{34}H_{34}ON_2S \cdot 2HCl$. *Duplodibenzylidenacetoneoxytrisulphide*, $C_{34}H_{32}OS_3$, m. p. $110-127^\circ$ (decomp.), is obtained by saturating an alcoholic suspension of dibenzylidenacetone with hydrogen sulphide, adding a slight excess of alcoholic ammonia, and again passing an excess of hydrogen sulphide. If the solution is saturated with ammonia and submitted to the prolonged action of hydrogen sulphide, *duplodibenzylidenacetonepentasulphide*, $C_{34}H_{32}S_5$, m. p. 201° , is obtained, which crystallises in colourless needles, and is oxidised by potassium permanganate and sulphuric acid in the presence of a little benzene to *duplodibenzylidenacetoneheptaoyltetrasulphide*, $C_{34}H_{32}O_4S_4$, decomp. $160-161^\circ$, and *duplodibenzylidenacetonehexaoxytetrasulphide*, $C_{34}H_{32}O_6S_4$, decomp. 130° .

The action of hydrogen chloride, followed by hydrogen sulphide, on an alcoholic suspension of dibenzylidenacetone yields a substance, $2C_{17}H_{17}O_3 \cdot 3H_2S$, decomp. 150° , or *triplodibenzylidenacetone tetrasulphide*, $C_{51}H_{50}S_4$, m. p. $99-100^\circ$.

Sperauski's thiomenthene, $C_{20}H_{34}S$, m. p. 50° (Abstr., 1907, i, 627), is precipitated when hydrogen sulphide is passed into a 10% alcoholic solution of menthone saturated with hydrogen chloride.

C. S.

Study of the Benzoin Condensation. THOR EKECRANTZ and ALFR. AHLQVIST (*Arkiv. Kem. Min. Geol.*, 1908, 3, No. 13, 1-26).

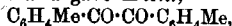
—The benzoin condensation is not general with aromatic aldehydes, the possibility of the reaction depending on the nature of the substituent groups present. The presence of amino-, hydroxy-, or

nitro-groups, or of large side-chains, in the aldehyde molecule hinders the reaction, whilst the presence of the substituent group in the meta-position is most favourable to the condensation. With aldehydes containing alkyl- or alkyloxy-groups, the reaction, in general, soon results in an equilibrium between condensation product and unchanged aldehyde. As a secondary reaction, occurs the formation of the acid corresponding with the aldehyde. With nitro-substituted benzaldehydes, large quantities of the acids are formed instead of benzoin, and in the case of *m*- and *p*-nitrobenzaldehydes the ethyl esters of the acids are also formed. With volatile acids, the corresponding cyanohydrins are often formed. Since, in the authors' condensation experiments, the alcohol corresponding with the aldehyde, and formed by the simultaneous oxidation and reduction of the aldehyde, was in no case detected, it is assumed that this alcohol takes part in various reactions, resulting in the formation of resin, which always arises in smaller or larger amount in the reaction between potassium cyanide and substituted aromatic aldehydes. Without invalidating Bredig and Stern's hypothesis that the benzoin condensation is due to catalysis by cyanogen ions (*Zeitsch. Elektrochem.*, 1904, 10, 582—587), the explanation of the varying behaviour of aromatic aldehydes when treated with potassium cyanide may be found in the acid character of the aldehydes (compare Euler, Abstr., 1906, i, 140). In the case of all aldehydes with which the degree of dissociation is greater than with hydrogen cyanide, for example, the nitrobenzaldehydes, aldehyde salt will be obtained in relatively large quantity, hydrogen cyanide, which is only slightly dissociated, being formed at the same time. Since potassium cyanide in aqueous solution is not only electrolytically dissociated, but also hydrolysed, according to the equation: $\text{KCN} + \text{H}_2\text{O} = \text{KHO} + \text{HCN}$, the formation of the acid corresponding with the aldehyde may be simply explained as follows: (1) under the influence of dilute alkali a hydrogen atom migrates from one aldehyde molecule to another, giving, for example, $\text{C}_6\text{H}_4\text{Br}\cdot\text{CH}(\text{OH})\cdot$ and $\text{C}_6\text{H}_4\text{Br}\cdot\text{CO}\cdot$; (2) these residues take up respectively the hydrogen and hydroxyl formed by hydrolysis, giving $\text{C}_6\text{H}_4\text{Br}\cdot\text{CH}_2\cdot\text{OH}$ and $\text{C}_6\text{H}_4\text{Br}\cdot\text{CO}_2\text{H}$. When these acids are formed in large quantities, they give rise to their strongly dissociated potassium salts, which cause a diminution of the cyanogen ions and so prevent the benzoin condensation from taking place. The condensation can therefore only occur with aldehydes which only exhibit a slight tendency to form salts, such as benzaldehyde and its alkyl and alkyloxy-derivatives and halogen derivatives of benzaldehyde and furfuraldehyde. In cases where the substituent group does not alter appreciably the degree of dissociation of the aldehyde, the condensation is considerably retarded by the presence of a side-chain.

In their experiments on the benzoin reaction, the authors dissolved 10—20 grams of aldehyde in four times the quantity of 66% alcohol and heated the mixture, together with an amount of potassium cyanide equal to 10—15% of that of the aldehyde, either (1) in a reflux apparatus on the water-bath for one hour, or (2) in a sealed tube at 60°.

With benzaldehyde, benzoin, benzoic acid, and mandelonitrile were formed.

Of the three tolualdehydes, the para-compound is the slowest to react and the meta-compound the most ready. From *p*-tolualdehyde, *p*-toluic acid is obtained along with the *p*-toluoin. *o*-Toluoic, $C_{10}H_8O_3$, crystallises from aqueous alcohol in silky needles, m. p. 79° . *m*-Toluoic could only be obtained as a syrup, oxidation of which with chromic oxide in acetic acid gave *m*-tolil,



which crystallises from alcohol in yellow needles, m. p. 103° .

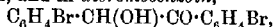
Cuminaldehyde yields cuminoic and cuminic acid. With salicylic aldehyde, no benzoin condensation occurs, the aldehyde undergoing profound decomposition with formation of resin (compare Zincke, *Annalen*, 1883, 216, 316). *p*-Hydroxybenzaldehyde and vanillin remain almost entirely unchanged, whilst anisaldehyde yields anisoic and anisic acid. Experiments made with 3-methoxy-4-benzyloxybenzaldehyde in order to ascertain the influence of side-chains on the reaction, showed that this compound yields no benzoin.

Benzanisoin, $OH \cdot CHPh \cdot CO \cdot C_6H_4 \cdot OMe$, which was prepared by the action of potassium cyanide on a mixture of benzaldehyde and anisaldehyde in 80% alcohol, and is analogous to Fischer's benzo-furoin (*Abstr.*, 1880, 798), crystallises from aqueous alcohol in colourless needles, m. p. $105-106^\circ$, and dissolves in most organic solvents.

Cuminanisoin, $C_6H_4Pr^s \cdot CH(OH) \cdot CO \cdot C_6H_4 \cdot OMe$, separates from aqueous alcohol in crystals, m. p. $81-82^\circ$, and is readily soluble in the ordinary organic solvents.

m-Chlorobenzaldehyde yields small quantities of *m*-dichlorobenzoin, together with *m*-chlorobenzoic acid.

m-Bromobenzaldehyde yields *m*-bromobenzaldehyde cyanohydrin, *m*-bromobenzoic acid, and *m*-dibromobenzoin,



which crystallises from aqueous alcohol in pale yellow needles, m. p. $123-124^\circ$, and dissolves readily in the ordinary organic solvents.

From *m*-aminobenzaldehyde, no definite product could be obtained.

T. H. P.

Preparation of Derivatives of Benzanthrone. BADISCHE ANILIN- & SODA-FABRIK (D.R.-P. 200335).—The alkyl derivatives of anthraquinone and α -aminoanthraquinone can be employed in the production of derivatives of benzanthrone (compare *Abstr.*, 1907, i, 324, 943). β -Methylanthranol, produced by the reduction of β -methylanthraquinone with tin and hydrochloric acid, when heated at $140-150^\circ$ with glycerol and concentrated sulphuric acid gives rise to *methylbenzanthrone*, yellow needles, m. p. 199° .

Dimethylbenzanthrone, yellow needles, m. p. 165° , is similarly obtained from 1 : 3-dimethylanthraquinone (m. p. $159-163^\circ$), the latter being first converted into the corresponding oxanthranol. G. T. M.

Constitution and Colour. II. FRIEDRICH KEHRMANN (*Ber.*, 1908, 41, 3396. Compare this vol., i, 699).—A criticism of K. H. Meyer's views on the halochromism of quinones (this vol., i, 731). J. J. S.

Preparation of Chloroacylaminoanthraquinones. BADISCHE ANILIN- & SODA-FABRIK (D.R.-P. 199758).—The aminoanthraquinones when chlorinated or brominated give rise to polyhalogenated derivatives, but when chlorine or chlorine evolving agents react with the acylaminoanthraquinones, the monochloro-derivatives are produced containing the halogen in the ortho- or para-position with regard to the amino-group.

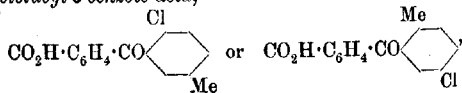
4-Chloroacetyl-1-aminoanthraquinone, yellow crystals, m. p. 203—204°, obtained by passing chlorine into a glacial acetic acid solution of acetyl-1-aminoanthraquinone and sodium acetate at 80°, gives, on hydrolysis with sulphuric acid, 4-chloro-1-aminoanthraquinone, red needles, m. p. 179—180°. 4:8-Dichloro-1:5-diacetyldiaminoanthraquinone, brown crystals, m. p. above 300°, and 4:8-dichloro-1:5-diaminoanthraquinone, red needles with green reflex, were similarly obtained.

1:5-Anthraquinonyldiurethane, from 1:5-anthraquinone and ethyl chlorocarbonate, when chlorinated in glacial acetic acid gives rise to 4:8-dichloro-1:5-anthraquinonyldiurethane, brown crystals. The preparation of 1-chloro-2-acetylaminoanthraquinone, yellow leaflets or needles, m. p. 240—241°, 1-chloro-2-aminoanthraquinone, reddish-brown crystals, m. p. 234—236°, and 1-chloro-2-anthraquinonyldiurethane, yellow leaflets m. p. 189—191°, is carried out in a similar manner. G. T. M.

Friedel and Crafts' Reaction. GUSTAV HELLER and KURT SCHÜLKE (*Ber.*, 1908, 41, 3627—3638).—The authors find that in the condensation of phthalic anhydride and benzene in presence of aluminium chloride, a reaction previously studied by Heller (*Zeitsch. angew. Chem.*, 1906, 19, 669), exactly 1 mol. of aluminium chloride, Al_2Cl_6 , is required for the condensation of 1 mol. of phthalic anhydride, and suggest that an intermediate compound is formed, thus: $C_6H_4O_2 + Al_2Cl_6 + C_6H_6 \rightarrow C_8H_4O_2 \cdot Al_2Cl_6 \cdot C_6H_6 \rightarrow C_{14}H_8O_2 \cdot Al_2Cl_6 + HCl$. This reaction has also been extended to the case of other hydrocarbons and some of their derivatives. From benzene and toluene, almost quantitative yields of *o*-benzoylbenzoic and *p*-toluoyl-*o*-benzoic acids were obtained. When the latter is heated on the water-bath with fuming sulphuric acid (20% SO_3), β -methylanthraquinone is obtained in almost colourless needles (Limpricht and Wiegand, *Abstr.*, 1900, i, 498, state that the crystals are usually yellow).

A 97% yield of α -naphthoyl-*o*-benzoic acid is obtained from naphthalene when benzene is used as solvent, and the acid is condensed to naphthanthraquinone by warming with 10 parts of concentrated sulphuric acid at 45—50° (compare Elbs, *Abstr.*, 1886, 1037). This yields a monosulphonic acid on treatment with fuming sulphuric acid (20% SO_3). Anthracylbenzoic acid is formed from anthracene, benzene being used as a solvent, but it could not be isolated in the pure state.

p-Chlorotoluoyl-*o*-benzoic acid,



prepared from *p*-chlorotoluene, crystallises in aggregates of slender needles, m. p. 164—165°. When warmed with fuming sulphuric acid,

it condenses to 4-chloro-1-methylantraquinone, which forms long, slender, yellow needles, m. p. 164°; on oxidation with permanganate, the acid yields p-chlorocarboxybenzoyl-o-benzoic acid, $C_{15}H_9O_5Cl$, a crystalline powder, m. p. 234—236°.

When 4-chloro-1-methylantraquinone is heated under pressure with 66% nitric acid to 195°, it yields 4-chloroantraquinone-1-carboxylic acid, crystallising in bundles of yellow needles, m. p. 228—229°.

o-Chlorotoluoyl-o-benzoic acid, the exact constitution of which was not determined, is prepared from o-chlorotoluene; it forms crystalline aggregates, m. p. 182—183°, and on warming with fuming sulphuric acid gives chloromethylantraquinone (Me:Cl=1:2 or 2:3), which forms glistening needles, m. p. 213°. By oxidising o-chlorotoluoyl-o-benzoic acid with permanganate, o-chlorocarboxybenzoyl-o-benzoic acid, $C_{15}H_9O_5Cl$, is produced. This forms long rhombohedra, and loses H_2O above 110°, sinters at 184°, and melts at 188°.

When the chloromethylantraquinone just described is heated under pressure with 66% nitric acid at 210°, β -chloroantraquinone-carboxylic acid ($CO_2H:Cl=1:2$ or $2:3$) is formed. This crystallises in slender, pale yellow, bushy needles, m. p. 280°; the sodium salt forms almost colourless needles. On heating above its m. p., the acid loses carbon dioxide, and 2-chloroantraquinone is left. J. C. C.

The Colour and Affinity for Mordants of Anthraquinone Derivatives. GUSTAV HELLER (*Ber.*, 1908, 41, 3639—3640).— β -Chloroantraquinonecarboxylic acid and 4-chloroantraquinone-1-carboxylic acid (preceding abstract), like the other simple derivatives of anthraquinone, are pale yellow. The alkaline solutions, however, are almost colourless, and the sodium salts are even paler than the acids. The substances have no affinity for mordants, and it may generally be stated that the affinity for mordants of anthraquinone derivatives is proportional to the intensity of colour of the sodium salts. J. C. C.

Phenanthrene Series. XXIV. Morpholquinone from Phenanthrene. JULIUS SCHMIDT and JULIUS SÖLL (*Ber.*, 1908, 41, 3696—3703).—Morpholquinone, 3:4-dihydroxyphenanthraquinone, may be obtained from 3-hydroxyphenanthraquinone (Werner, *Abstr.*, 1902, i, 440) by the following stages: nitration, reduction, diazotisation, and boiling with water. A modification of Werner's method of diazotising 3-aminophenanthraquinone is described. Nitric acid reacts with 3-hydroxyphenanthraquinone, yielding a mixture of the isomeric 2- and 4-nitro-derivatives, both of which dissolve readily in sodium carbonate solution. One of these crystallises from glacial acetic acid in yellow needles, and is identical with Werner's product. It has been found possible to obtain a dinitro-derivative by the action of nitric acid on 3-hydroxyphenanthraquinone. It crystallises in yellow needles, m. p. 227—228°, and reacts with o-phenylenediamine, yielding a phenanthraphenazine, m. p. 249°. The morpholquinone is obtained, together with other products, when the mixture of mononitro-derivatives is reduced with tin and hydrochloric acid, the resulting mixture of amino-compounds diazotised, and the diazo-solution boiled.

Morpholquinone can also be prepared from the mother liquors from 3-nitrophenanthraquinone, obtained when 9:10-diaminophenanthrene is treated with acetic anhydride and nitric acid (see following abstract). The mother liquor is evaporated, reduced and diazotised, and the resulting diazo-solution boiled.

J. J. S.

Phenanthrene Series. XXIII. 3-Nitrophenanthraquinone and its Derivatives. JULIUS SCHMIDT and JULIUS SÖLL (*Ber.*, 1908, 41, 3679—3695. Compare Werner, *Abstr.*, 1904, i, 863; Schmidt and Kämpf, *Abstr.*, 1902, i, 757, 797).—Starting with 3-nitrophenanthraquinone, it has been found possible to prepare morpholquinone (compare preceding abstract). A convenient method for the preparation of the 3-nitro-derivative has been worked out, and numerous derivatives have been prepared.

In the case of 3-nitrophenanthraquinonedioxime, it has been found that the replacement of the hydrogen atoms of the oximino-groups by methyl or acetyl produces a weakening in colour (*Abstr.*, 1907, i, 630, 1054), and the same change is observed during anhydride formation (this vol., i, 16).

A modification of Pschorr's method (*Abstr.*, 1902, i, 672) for the preparation of 9:10-diaminophenanthrene hydrochloride is described. This salt is best nitrated by first acetylating by boiling with a mixture of acetic anhydride, acetic acid, and the theoretical amount of sodium acetate, and then stirring and heating with concentrated nitric acid in the presence of acetic acid, acetic anhydride, and a small amount (1 gram) of copper. The yield of pure 3-nitrophenanthraquinone is only some 20% of the theoretical.

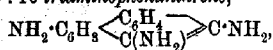
3-Nitrophenanthraquinone monoxime, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \cdot \begin{smallmatrix} \text{C}_6\text{H}_4 \\ \diagup \quad \diagdown \\ \text{C} \cdot \text{NH} \end{smallmatrix} \text{CO}$, obtained by shaking 3-nitrophenanthraquinone with alcoholic ammonia for five hours, crystallises from benzene in green prisms with a brassy lustre, and decomposes at 203°. Its benzene solution with hydrogen chloride yields a red *hydrochloride*. When boiled with acetic acid, it is converted back into the quinone.

3-Nitrophenanthraquinone semicarbazone, $\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}_4$, is extremely unstable, and has m. p. 254° (decomp.). The dioxime, $\text{C}_{14}\text{H}_9\text{O}_4\text{N}_4$, crystallises from alcohol in yellowish-red needles, m. p. 200° (decomp.). In the preparation of oximes of nitro-derivatives, it has been found advisable to use granular witherite to neutralise the liberated hydrogen chloride.

The *diacetyl* derivative of the dioxime, $\text{C}_{15}\text{H}_{13}\text{O}_6\text{N}_4$, crystallises from benzene in pale yellow plates, m. p. 183° (decomp.). The anhydride of the dioxime, **3-nitrophenanthrafurazan**, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \cdot \begin{smallmatrix} \text{C}_6\text{H}_4 \\ \diagup \quad \diagdown \\ \text{C} \cdot \text{N} \end{smallmatrix} \text{C} \cdot \text{N} > \text{O}$, is obtained when the dioxime is shaken with benzoyl chloride and sodium hydroxide solution. It crystallises from alcohol in yellow needles, m. p. 230—231°.

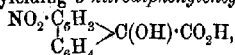
The *dimethyl ether* of the dioxime, $\text{C}_{16}\text{H}_{14}\text{O}_4\text{N}_4$, obtained by the action of methyl sulphate and concentrated sodium hydroxide solution on a benzene solution of the oxime, crystallises from alcohol in pale yellow needles, m. p. 190—192°.

When reduced with an alcoholic solution of stannous chloride, the dioxime yields 3 : 9 : 10-triaminophenanthrene,



which is transformed into a *hexa-acetyl* derivative, $\text{C}_{26}\text{H}_{26}\text{O}_6\text{N}_3$, m. p. 307° , when heated with acetic anhydride and sodium acetate.

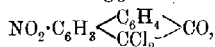
Warm 10% potassium hydroxide solution reacts with 3-nitrophenanthraquinone, yielding 3-nitrodiphenyleneglycolic acid,



m. p. $239-240^\circ$, and 3-nitrofluorenone, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \begin{array}{c} \text{C}_6\text{H}_3 \\ \text{C}_6\text{H}_4 \end{array} \text{C}=\text{O}$, which crystallises from alcohol in yellowish-brown needles, m. p. $209-210^\circ$. The

amine, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \begin{array}{c} \text{C}_6\text{H}_3 \\ \text{C}_6\text{H}_4 \end{array} \text{C}:\text{N} \cdot \text{OH}$, also forms yellowish-brown needles, m. p. 240° .

Phosphorus pentachloride reacts with 3-nitrophenanthraquinone, yielding equal quantities of 9 : 9-dichloro-3-nitro- and 10 : 10-dichloro-3-nitrophenanthrones, $\text{NO}_2 \cdot \text{C}_6\text{H}_3 \begin{array}{c} \text{C}_6\text{H}_3 \\ \text{CO} \end{array} \text{C} \begin{array}{c} \text{C}_6\text{H}_4 \\ \text{CO} \end{array} \text{C} \text{Cl}_2$ and



which can be separated by means of benzene; the α -compound separates in pale yellow plates, m. p. $191-193^\circ$. The isomeric β -compound has m. p. $143-145^\circ$. When reduced with tin and hydrochloric acid, these two compounds yield the corresponding chloroaminohydroxyphenanthrenes, $\text{NH}_2 \cdot \text{C}_6\text{H}_3 \begin{array}{c} \text{C}_6\text{H}_3 \\ \text{C}(\text{OH}) \end{array} \text{C} \text{Cl}$ and $\text{NH}_2 \cdot \text{C}_6\text{H}_3 \begin{array}{c} \text{C}_6\text{H}_3 \\ \text{CCl} \end{array} \text{C} \cdot \text{OH}$, which have been isolated in the form of their hydrochlorides. All attempts to nitrate these chlorohydroxy-compounds proved unsuccessful.

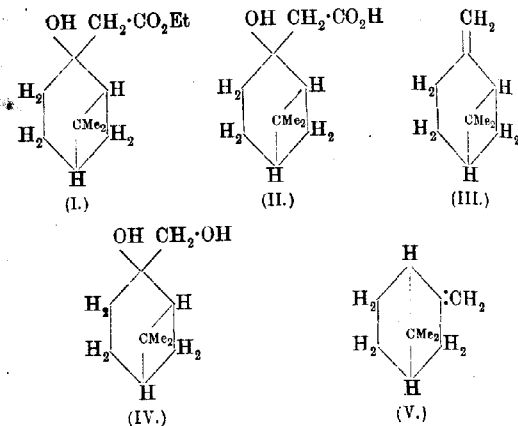
3-Aminophenanthraquinone (Werner, Abstr., 1902, i, 441) yields a *monoxime*, $\text{C}_{14}\text{H}_{10}\text{O}_2\text{N}_2$, which crystallises in small, reddish-brown prisms, m. p. 247° (decomp.).

Attempts to obtain nitro-derivatives of the amine proved unsuccessful. With a mixture of acetic acid, acetic anhydride, and nitric acid, the base yields 3-aminodinitrodiphenic acid, $\text{C}_{14}\text{H}_8\text{O}_6\text{N}_3$, which crystallises from water in colourless plates, m. p. $200-201^\circ$ (decomp.).

J. J. S.

Terpenes and Ethereal Oils. XCVII. Synthesis of Fenchene, β -Pinene, Camphene, and Camphor from Nopinone. OTTO WALLACH (*Annalen*, 1908, 363, 1-19. Compare this vol., 813).—A previous attempt to prepare β -pinene from nopinone led to the synthesis of a hydrocarbon isomeric with the desired product (Abstr., 1907, i, 1058). Further experiments, however, have now resulted, not only in the synthesis of β -pinene, but also in the discovery of a remarkable intramolecular transformation. The interaction of nopinone, ethyl bromoacetate, and zinc leads to the formation of a hydroxy-ester (I). This was heated directly with potassium hydrogen

sulphate, and the resulting unsaturated ester hydrolysed. The terpene, obtained on distillation of the unsaturated acid, is now found to be identical with *Ld*-fenchene (V), which is obtained together with *Ll*-fenchene from *l*-fenchone in the same manner as *Dd*- and *Dl*-fenchenes are formed from *d*-fenchone (Abstr., 1899, i, 65; this vol., i, 809).



Ld-Fenchene, on oxidation, yields hydroxyfenchenic acid, m. p. 154°, and on hydration *isofenchyl* alcohol (Abstr., 1907, i, 1058), m. p. 62—63°, b. p. 202—204°, which on oxidation is converted into *isofenchone* (compare this vol., i, 809). *isofenchocamphoric* acid, m. p. 158—159°, has $[\alpha]_D + 12^\circ$.

On repeating the synthesis from nopinone, there was obtained *Ll*-fenchene, which resembles the *Ld*-isomeride and on hydration forms *isofenchyl* alcohol, but on oxidation yields hydroxyfenchenic acid, m. p. 135—136°. It is found that the nature of the end-product depends on the manner in which water is split off from ethyl nopinolacetate (I). If the dehydrating agent employed is acetic anhydride, the end-product is a mixture of fenchene and β -pinene, the latter being obtained in greatest amount if the ester is hydrolysed before the dehydration.

Nopinolacetic acid (II), formed by hydrolysis of its ester by means of alkalis, crystallises from light petroleum in prisms, m. p. 83—85°, and is stable towards lead dioxide, but is readily attacked by boiling dilute sulphuric acid, yielding *isofenchyl* alcohol and an acid substance. When heated with potassium hydrogen sulphate, nopinolacetic acid is converted into an isomeric *acid*, m. p. 85—86°, and a volatile acid, m. p. 174—175°, which may be homodihydrocuminic acid. The action of acetic anhydride on nopinolacetic acid leads to the formation of two unsaturated acids, one of which loses carbon dioxide during the reaction and forms *l*- β -pinene (III), b. p. 163—164°, D^{22}_D 0.8660—0.8675, n^{25}_D 1.4724—1.4749, $\alpha - 23^\circ 5'$ to $-22^\circ 20'$; this on oxidation

with permanganate forms nopic acid identical with that obtained from natural β -pinene. The second acid is more stable, and decomposes only on distillation, yielding fenchene.

A sparingly soluble *glycol* (IV), crystallising in leaflets, m. p. 75–77° (compare Wagner, Abstr., 1894, i, 610), is obtained together with nopic acid by careful oxidation of the synthetical, as also of the natural, β -pinene. When heated with dilute acids, it yields an *aldehyde*, which has an odour of cuminol, forms a *semicarbazone*, and reduces silver solutions with formation of a silver mirror. When heated at 60° with glacial acetic-sulphuric acid, β -pinene yields terpinene, b. p. 175–180°, and small amounts of *isofenchyl acetate*, which suggests the presence of traces of fenchene in the synthetical pinene.

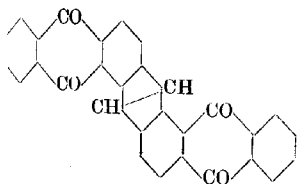
The action of hydrogen chloride on synthetical β -pinene in cooled glacial acetic acid, or anhydrous ethereal solution, leads to the formation of bornyl chloride, m. p. 117–120°, $[\alpha]_D - 22.75^\circ$, and dipentene dihydrochloride, m. p. 49–50°.

Camphene, formed from the synthetical *L*-bornyl chloride by the action of sodium acetate and glacial acetic acid at 190–200°, is converted by way of its acetate into *isoborneol*, which on oxidation with chromic acid yields camphor. This series of reactions constitutes the first synthesis of camphor from a compound (nopinone) containing a smaller number of carbon atoms.

It is considered that the first product of the dehydration of nopinol-acetic acid (II) is probably β -pinene (III), which undergoes transformation into *Ll*-fenchene, this finally changing into *Ld*-fenchene (V). Such a series of changes must entail a considerable intramolecular transformation, the mechanism of which remains to be investigated.

G. Y.

[Preparation of Complex Anthracene Derivatives.] BADISCHE ANILIN- & SODA-FABRIK (D.R.-P. 199756).— β -Methylanthraquinone, when heated at 150–170° with alcoholic potash, gives rise to a



condensation product, at first present as a dihydroanthracene derivative, but which on oxidation with alkali hypochlorite or atmospheric oxidation yields the annexed compound. This substance separates from hot nitrobenzene in golden-yellow crystals, not melting below 360°. The oxidation may also be

effected by adding sodium nitrate to the alcoholic potash; the reaction then takes place at 140–150°.

The halogenated derivatives of β -methylanthraquinone give similar condensation products.

G. T. M.

Preparation of Citral Hydrate. PIERRE COULIN (D.R.-P. 198483).—The dihydrodisulphonic derivative of citral was obtained when citral or lemon-grass oil was treated with aqueous sodium sulphite, dilute sulphuric acid being slowly added to neutralise the alkali hydroxide.

The liquid was then cooled to 0° , and treated with 50% sulphuric acid or 30% hydrochloric acid to form an additive compound, from which citral hydrate can be regenerated by the action of alkali hydroxides.

G. T. M.

[Isolation of an Isomeride of Citral Hydrate.] PIERRE COULIN (D.R.-P. 198714. Compare preceding abstract).—The product obtained by the action of acids on the labile dihydrodisulphonic derivative of citral consists of citral hydrate, which is not volatile in steam, and an isomeric compound, which is readily volatile under these conditions. This isomeride of citral hydrate has the following properties: b. p. $93.2-95.2^{\circ}/13$ mm., $D_{25}^{20} 0.964$, $n_{25}^{20} 1.44991$, semicarbazone, m. p. 172° , and thiosemicarbazone, m. p. 129° .

When condensed with acetone or methyl ethyl ketone in the presence of alkali hydroxides, this isomeride of citral hydrate gives rise to condensation products, which, by heating with water or a hydrogen sulphite, or treatment with acid, yield perfumes having respectively the formulæ $C_{13}H_{22}O_2$ and $C_{14}H_{24}O_2$.

G. T. M.

[The Condensation of Ketones and Citral Hydrate.] PIERRE COULIN (D.R.-P. 200654).—The condensation of citral hydrate with acetone or methyl ethyl ketone in the presence of sodium peroxide or potassium hydroxide leads to the production of ψ -ionone hydrate and methyl- ψ -ionone hydrate respectively.

G. T. M.

Preparation of Neutral Esters from Sandal Wood Oil. KNOLL & Co. (D.R.-P. 201369).—*Santolyl succinate* was prepared either by mixing sandal wood oil and succinyl chloride in the presence of pyridine, or by heating the oil with phenyl succinate at 200° in the presence of sodium hydroxide.

Santolyl phosphate is prepared from sandal wood oil, triphenyl phosphate, and sodium hydroxide at 200° . The santolyl esters of other polybasic acids, such as phthalic, citric, and methylenecitric acids, are obtained by similar means.

G. T. M.

Identity of Illicyl Alcohol and α -Amyrin. ÉMILE JUNGLEISCH and HENRI LEROUX (*Compt. rend.*, 1908, 147, 862–864*).—The specimen of illicyl alcohol obtained by Personne from the bird-line yielded by the common holly, to which the formula $C_{25}H_{41}O$ was ascribed (Abstr., 1884, 1365), formed colourless needles, m. p. 175° . After several recrystallisations from alcohol, however, it has m. p. $181-181.5^{\circ}$, $[\alpha]_D +91.8^{\circ}$ (α -amyrin has $181-181.5^{\circ}$ and $\alpha +91.6^{\circ}$ respectively), and the results of its analysis agree closely with the formula, $C_{30}H_{50}O$, of α -amyrin. Further, the acetate prepared either from illicyl alcohol or from α -amyrin has m. p. 220° and $[\alpha]_D +77.9^{\circ}$ or $+77.0^{\circ}$, whilst the benzoate has m. p. 192° and $[\alpha]_D +93.1^{\circ}$ or $+93.58^{\circ}$, according to the source of preparation.

The identity of the two benzoates is, moreover, rendered conclusive by the fact that both are dimorphous. The acetone solution deposits the benzoate, sometimes separately, sometimes simultaneously, in the form of very long, silky needles, m. p. 170° (on Maquenne block), and

* and *J. Pharm. Chim.*, 1908, 28, 481–486.

as prisms with brilliant facets, m. p. 192° (on Maquenne block). If cooled with one form, the solution deposits the whole crop in that form, but both can exist together in solution for several days. The needles, if cooled after melting at 170° , re-melt at 192° (on Maquenne block), which is also the m. p. of the needles if previously heated at 110° for fifteen hours. The needle crystals, if slowly heated, have m. p. 192° , and if melted at 170° , resolidify on continued heating and again melt at 192° . Wyruboff finds that the prismatic benzoates of the two origins are optically identical.

E. H.

Bakankosin. ÉMILE BOURQUELOT and HENRI HÉRISSEY (*Compt. rend.*, 1908, 147, 750—752; *J. Pharm. Chim.*, 1908, [vi], 28, 433—439).—*Strychnos Bakanko*, from which the authors have isolated (Abstr., 1907, i, 33C) the glucoside bakankosin, has been shown by Jumelle to be identical with Baillon's *Strychnos Vacacoua*. The fruit originally employed by the authors was not ripe; they have now examined the seeds from the ripe plant, and find that they also contain the new glucoside, although in less amount (0.92% instead of the 3.6% from the unripe seeds). One part of anhydrous bakankosin dissolves in 3164 parts of ethyl acetate (anhydrous), 55 parts of 95% alcohol, 12 parts of water, or 4 parts of methyl alcohol. Analysis and a cryoscopic determination of the molecular weight show that the glucoside has the formula $C_{16}H_{23}O_8N_2H_2O$. Bakankosin is not racemised by baryta, thus being unlike other nitrogenous glucosides. The equation $C_{16}H_{23}O_8N_2 + H_2O = C_6H_{12}O_6 + C_{10}H_{11}O_8N_2$ is provisionally proposed to represent the hydrolysis effected by emulsin.

Neither the shell of the seed nor the pulp of the fruit contains any glucoside hydrolysed by emulsin.

E. H.

Preparation of a Derivative of Gallocyenin. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. (D.R.-P. 198508).—*m*-Chloro-*p*-nitrosodiethylaniline, m.p. $76-77^{\circ}$, a green base, dissolves in alcohol to a yellow solution; its hydrochloride, when condensed with gallaminic acid, gives rise to a gallocyenin derivative analogous to celestin-blue, but of a greener shade.

G. T. M.

Salts of Guanidine, Dicyanodiamide, and Melamine with Acid Dyes. LEOPOLD RADLBERGER (*Monatsh.*, 1908, 29, 937—950. Compare Suida, Abstr., 1907, ii, 112).—Guanidine, dicyanodiamide, and melamine behave as monoacidic bases towards the following acid dyes: crystal-ponceau, $C_{20}H_{14}O_7N_2S_2$; ponceau 2G, $C_{16}H_{12}O_7N_2S_2$; chromotrope 2R, $C_{16}H_{12}O_8N_2S_2$; orange IV, $C_{18}H_{16}O_8N_2S_2$, and orange II, $C_{16}H_{12}O_4N_2S$. The salts formed are analogous to ammonium salts, and are obtained, as a rule, in a crystalline form. The following salts were prepared: guanidine with crystal-ponceau, $C_{23}H_{21}O_7N_5S_2H_2O$, small, reddish-brown needles; with ponceau 2G, $C_{18}H_{16}O_7N_5S_2$, red powder; with chromotrope 2R, $C_{18}H_{16}O_8N_5S_2$, small, short, brown needles; with orange II, $C_{17}H_{17}O_4N_5S$, yellowish-orange leaflets; with orange IV, $C_{19}H_{20}O_8N_5S$, yellow, monoclinic leaflets.

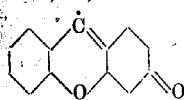
Dicyanodiamide with crystal-ponceau, $C_{21}H_{19}O_7N_7S_2 \cdot 2H_2O$, small, brownish-red needles; with ponceau 2G, $C_{20}H_{20}O_7N_7S_2$, red powder; with chromotrope 2R, $C_{20}H_{20}O_8N_7S_2$, brown powder; with orange

II, $C_{18}H_{16}O_4N_6S$, orange, irregular leaflets; with orange IV, $C_{26}H_{18}O_8N_8S$, orange-yellow prisms.

Melamine with crystal-ponceau, $C_{28}H_{20}O_4N_4S_2$, ref., crystalline powder; with orange II, $C_{18}H_{16}O_4N_6S$, small, pale yellow crystals.

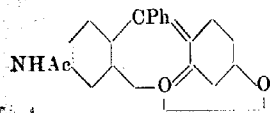
W. H. G.

Carboxonium Dyes. I. The Chromogen of Fluorescein and the Simplest Rosamine. FRIEDRICH KEHRMANN and O. DÜGLER (*Ber.*, 1908, 41, 3440—3447).—Following Möhlau and Koch (Abstr., 1895,



i, 46), the authors give the name "fluorone" to the annexed group, but prefer the ortho-quinonoid structure depicted in the annexed formulae.

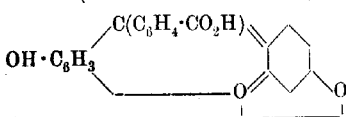
When *m*-acetylaminophenol and benzotrichloride are heated at 150—160° in the presence of nitrobenzene, several substances are obtained, from which, after somewhat complicated treatment, two acetylated bases are isolated and separated from each other by alcohol. The less soluble one is *acetylaminophenylfluorone* (annexed constitution), m. p. above



360°, which crystallises in tufts of red needles, is insoluble in alkalis, and gives strongly fluorescent solutions in alcohol, glacial acetic acid, benzene, or warm dilute mineral acids. In concentrated sulphuric acid it gives a yellow solution with a strong green fluorescence, which remains unchanged by the addition of ice. By hydrolysis by moderately concentrated hydrochloric acid and boiling alcohol, it yields the chloride of the base, which crystallises in red leaflets with a blue reflex. The base, $NH_2 \cdot C_6H_5 \cdot \langle CPh \rangle \cdot C_6H_5 \cdot O$, liberated by excess

of ammonium hydroxide, has m. p. 305°, crystallises in dark red needles, forms orange-red salts, and by the elimination of the amino-group yields *phenylfluorone*, m. p. 204°, which forms orange-yellow crystals, is insoluble in alkalis or dilute mineral acids, and gives fluorescent solutions. The substance is the chromogen of fluorescein, and shows a striking analogy to *aposafranone*, *oxazone*, and *thiazone*.

Hydroxyphenylfluorone, obtained by replacing the amino-group of the preceding base by hydroxyl, is identical with Doebner's resorcinolbenzen (*Annalen*, 1878, 217, 234), and shows a remarkable similarity



in its colour phenomena to fluorescein, which, as its carboxylic acid, must have the annexed constitution (compare R. Meyer and Hoffmeyer, Abstr., 1892, 970.)

The other product isolated from the substances obtained from *m*-acetylaminophenol and benzotrichloride is *diacetylphenorosamine*, the chloride of which, $NHAc \cdot C_6H_5 \cdot \langle CPh \rangle \cdot C_6H_5 \cdot NHAc$, forms a yellow, fluorescent solution in hot water without hydrolysis. By treatment with ammonium hydroxide, the chloride is changed to a magenta-red, unstable colour base, which is spontaneously transformed to a colourless,

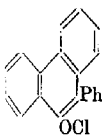
stable form, from which dilute acids regenerate yellow salts. By hydrolysis, the simplest rosamine is obtained as a yellowish-red, extremely fluorescent dye. C. S.

Quinonoid Ester Salts of Fluoran. ARTHUR G. GREEN and PERCY E. KING (*Ber.*, 1908, 41, 3434—3440. Compare Abstr., 1906, i, 670; 1907, i, 933).—In support of their ortho-quinonoid formula for the oxonium salts of quinolphthalein derivatives, the authors have prepared similar oxonium salts of the esters of fluoran and of 2:7-dimethylfluoran. The substances are unstable and extremely soluble, and their isolation is only possible in the form of the tin or zinc double chlorides. When hydrogen chloride is passed into hot methyl alcohol containing fluoran and stannic chloride, orange, prismatic crystals of the *stannichloride*, $\left[\text{CO}_2\text{Me} \cdot \text{C}_6\text{H}_4 \cdot \text{C} \begin{array}{c} \text{C}_6\text{H}_4 \\ \text{C}_6\text{H}_4 \end{array} \text{OCl} \right]_2 \cdot \text{SnCl}_4$, are obtained on keeping. The corresponding *stannichloride* of the ethyl ester crystallises in large, yellow prisms, containing 1 mol. EtOH. The *stannichloride* of the oxonium chloride of the methyl ester of 2:7-dimethylfluoran contains equal molecular quantities of the oxonium and of the metallic chloride; this appears to depend on the solubility, for zincchlorides containing one or two mols. of the oxonium chloride to one mol. of zinc chloride have been prepared, namely,

$\text{C}_{21}\text{H}_{16}\text{OCl}(\text{CO}_2\text{Me})_2 \cdot \text{ZnCl}_2$
 and $[\text{C}_{21}\text{H}_{16}\text{OCl}(\text{CO}_2\text{Me})]_2 \cdot \text{ZnCl}_2$.
 2:7-Dichlorofluoran, $\text{C}_{20}\text{H}_{10}\text{O}_3\text{Cl}_2$, m. p. 256—258°, is obtained by condensing phthalic anhydride and *p*-chlorophenol with concentrated sulphuric acid at 150—160°. The halogen atoms are not readily displaced, and the substance, as also the corresponding dinitrofluoran, does not readily esterify under the preceding conditions. C. S.

Dibenzopyrronium. HERMANN DECKER and HEINRICH FELSER (*Ber.*, 1908, 41, 3755—3757. Compare Abstr., 1907, i, 1064).—It has been found possible to prepare derivatives of dibenzopyrronium, the oxygen isologue of phenanthridine.

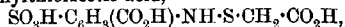
9-Phenyldibenzopyrronium may be obtained by the method of Bünzly and Decker (Abstr., 1904, i, 912), namely, by the action of magnesium phenyl bromide on diphenylmethylid (compare Graebe and Schestakoff, Abstr., 1895, i, 372). The *carbinol base* is not such a strong base as 9-phenyl-xanthonium carbinol; the *chloride* (annexed formula) dissolves in hydrochloric acid, forming a yellow solution without fluorescence; the *ferrichloride*, $\text{C}_{19}\text{H}_{13}\text{OCl}_2\text{FeCl}_3$, crystallises in lemon-yellow leaflets, m. p. 163°. W. H. G.



Preparation of 3-Oxythionaphthen. FARBERKE VORM. MEISTER, LUCIUS & BRÜNING (D.R.-P. 200200).—Methyl *o*-methylthiolbenzoate, $\text{SMe} \cdot \text{C}_6\text{H}_4 \cdot \text{CO}_2\text{Me}$, needles, m. p. 67°, volatile in steam, produced by alkylating *o*-methylthiolbenzoic acid, $\text{SMe} \cdot \text{C}_6\text{H}_4 \cdot \text{CO}_2\text{H}$, m. p. 168—169°, or the corresponding sodium, potassium, or calcium methylthiolbenzoate, when heated with sodium and potassium hydroxides (equal mols.) at 200° gives rise to 3-oxythionaphthen;

this condensation takes place even more readily when some sodium or potassium ethoxide is present in the fusion. G. T. M.

Preparation of a Dihydroxythionaphthencarboxylic Acid. FARBWERKE VORM. MEISTER, LUCIUS & BRÜNING (D.R.-P. 200202).—5-Sulphoanthranilylthiolacetic acid,



prepared from 5-sulphoaniline-2-carboxylic acid, yields a monopotassium salt; this compound when heated with alkali hydroxides, first at 130—140° and then at 160—185°, gives rise to a yellow fusion product, which, when dissolved in water and acidified, furnishes a dihydroxythionaphthen-2-carboxylic acid. On boiling with water, this acid yields a dihydroxythionaphthen, needles, m. p. 198°.

G. T. M.

[Preparation of Bases of the Adrenaline Series.] CHEMISCHE FABRIK AUF ACTIEN (VORM. E. SCHERING) (D.R.-P. 201245).—The acid reduction with sodium, potassium, or aluminium amalgams of 3:4-dihydroxyphenylglyoxime and the 3:4-dihydroxyphenylalkylglyoximes of the annexed general formula leads to the production of bases having a very intense hæmstatic action, but which are less toxic than adrenaline. Neither the bases nor their salts have yet been isolated in a pure state.



$\text{NOH}\cdot\text{C}\cdot\text{CR}\cdot\text{NOH}$

G. T. M.

Preparation of a Double Compound of Caffeine and Lithium Benzoate. PETER BERGELL (D.R.-P. 199108).—A crystalline double compound, $2\text{C}_8\text{H}_{10}\cdot\text{CO}_2\text{Li}\cdot\text{C}_6\text{H}_5\text{O}_2\text{N}_4$, is obtained in needles by evaporating below 50° under reduced pressure an aqueous solution of caffeine and lithium benzoate.

G. T. M.

Solubility of True and False Tannates of Quinine. F. MURARO (*Gazzetta*, 1908, 38, ii, 507—511).—A reply to Biginelli's criticisms (this vol., i, 562) on the author's previous paper (this vol., i 451).

T. H. P.

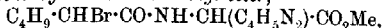
Preparation of α -Bromoisovalerylquinone. KNOLL & CO. (D.R.-P. 200063).—Quinine or its salts, when heated for three hours at 120° with α -bromoisovaleryl chloride or bromide, gives rise to α -bromoisovalerylquinine, an amorphous, yellow substance, dissolving in alcohols and in acids, but less soluble in benzene, toluene, light petroleum, or ether. From acid solution, it is precipitated by potassium mercuri-iodide.

G. T. M.

Synthesis of Polypeptides. XXVII. I. Derivatives of Histidine. EMIL FISCHER and LEE H. CONE (*Annalen*, 1908, 363, 107—117. Compare this vol., i, 773).—So far there has been described only one dipeptide, histidylhistidine, derived from histidine, which itself is widely spread amongst the natural proteins. It was of special interest, therefore, to undertake the synthesis of mixed peptides derived from this amino-acid, especially as these may be expected to occur amongst the degradation products of the proteins.

The combination of *l*-leucine with histidine is described in the present paper. As oxyhaemoglobin contains considerable quantities of *l*-leucine and histidine, which are present probably as a mixed dipeptide, and as *l*-leucyl-*l*-histidine is stable towards concentrated hydrochloric acid, it was hoped to find this dipeptide amongst the products of the action of concentrated hydrochloric acid on oxyhaemoglobin. In this the authors have been unsuccessful, as also, so far, in attempts to prepare the isomeride, *l*-histidyl-*l*-leucine. It is found that histidine hydrochloride is stable towards cold phosphorus pentachloride and acetyl chloride. Experiments with formylhistidine, which is described, are in progress.

d- α -Bromoisohexoyl-*l*-histidine methyl ester,



prepared from *d*- α -bromoisohexoyl chloride and histidine methyl ester (Fischer and Suzuki, Abstr., 1906, i, 73) in chloroform solution, crystallises from ethyl acetate in strongly refracting, colourless, hexagonal plates, m. p. 175° (corr.), dissolves in dilute mineral acids, and is reprecipitated by sodium carbonate.

d- α -Bromoisohexoyl-*l*-histidine, $C_{12}H_{18}O_3N_3Br$, prepared by hydrolysis of the ester with *N*-sodium hydroxide at the laboratory temperature and evaporation of the product after neutralisation with sulphuric acid, separates from water in crystals, m. p. 118° (corr.), has an acid reaction in aqueous solution, and gradually becomes yellow when dissolved in alkalis.

l-Leucyl-*l*-histidine, $C_4H_9 \cdot CH(NH_2) \cdot CO \cdot NH \cdot CH(C_4H_5N_3) \cdot CO_2H$, is best prepared by the action of 25% aqueous ammonia on the preceding bromo-compound at 25°. It separates from water in prisms or plates containing H_2O , loses H_2O at 100°/15–20 mm., when anhydrous has m. p. 178° (corr.) (decomp.), has a strong alkaline reaction in aqueous solution, gives a colourless precipitate with mercuric chloride solution and a red coloration with *p*-diazobenzenesulphonic acid,* and becomes dark coloured when boiled with dilute bromine water; these reactions show that the iminazole nucleus of the histidine is present unchanged in the dipeptide. The dipeptide is soluble in dilute acids and alkalis; the acid solutions give a heavy precipitate with phosphotungstic acid. The copper salt, $C_{12}H_{18}O_3N_3Cu \cdot 4H_2O$, formed by boiling the aqueous solution of the dipeptide with precipitated copper oxide, or by adding copper sulphate to the solution of the dipeptide in sodium hydroxide, is obtained in deep violet-blue crystals, which lose $4H_2O$, becoming lilac at 100°/15–20 mm. over phosphoric oxide, and dissolve in alcohol, forming a violet-blue solution. Whilst it is stable towards cold concentrated mineral acid, the dipeptide is hydrolysed completely in twenty-four hours by 20% hydrochloric acid at 100°, yielding *l*-leucine and *l*-histidine.

Formyl-*l*-histidine, $C_7H_9O_3N_3$, formed by heating histidine with anhydrous formic acid on the water-bath and evaporating the solution under 15–20 mm. pressure, crystallises from aqueous-methyl alcohol in slender needles, m. p. 203° (corr.), is readily soluble in water, slightly so in methyl alcohol, but almost insoluble in other organic solvents, has an acid reaction in aqueous solution, and gives a deep red coloration with *p*-diazobenzenesulphonic acid, which points to

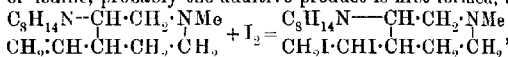
the formyl group as having entered into combination with the amino group of the histidine.

G. Y.

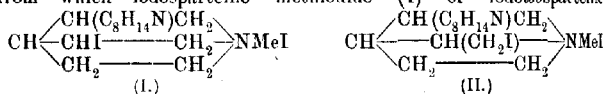
Action of Ozone on Thebaine. J. D. KIEDEL (D.R.P. 201324).—Thebaine hydrochloride in 10% aqueous solution is treated with ozonised oxygen at the ordinary temperature until the product is soluble in alkali hydroxides. The ethereal extract of the solution, when dried with potassium carbonate and concentrated, furnishes a compound, $C_{15}H_{21}O_5N$, which crystallises in needles or leaflets, m. p. 125–126°. This oxidation product still contains two methyl groups, and loses one of these by alkaline hydrolysis; it has probably the formula $C_{13}H_{15}ON(CO_2Me)(CO)OMe$. The substance is readily soluble in water, ethyl acetate, and alcohol.

G. T. M.

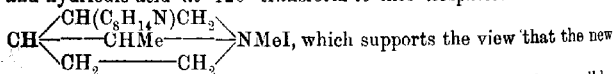
Sparteine. New Method of Ring-formation of α -Methylsparteine by the Action of Iodine. AMAND VALEUR (*Compt. rend.*, 1908, 147, 864–867).—Although the α -methylsparteine obtained by Moureu and Valeur (this vol., i, 44) by heating methylsparteinium hydroxide is certainly an unsaturated base, since it reduces a cold sulphuric acid solution of potassium permanganate, it is not reduced by sodium and ethyl or amyl alcohol, or by tin and hydrochloric acid. It combines with iodine, however, forming *di-iodo- α -methylsparteine*, $C_{15}H_{25}N_2MeI_2$, m. p. 177–178°, which does not behave as a simple additive product, has a very feebly alkaline reaction, and when treated in aqueous solution with freshly prepared silver oxide gives a strongly alkaline liquid, from which the di-iodide is re-formed by addition of potassium iodide. The latter observation indicates that the new compound is a quaternary ammonium iodide, and therefore must be the methiodide of an iodo-base. In the action of iodine, probably the additive product is first formed, thus:



from which iodosparteine methiodide (I) or iodoisosparteine



methiodide (II) can be formed according to which iodine atom wanders to the nitrogen. Whilst zinc and water or zinc and acetic acid reduce the di-iodide, giving α -methylsparteine only, phosphorus and hydriodic acid at 120° transform it into isosparteine methiodide,



di-iodide should be represented by formula (II). It is possible, however, that the isosparteine methiodide is formed by isomeric change from the α -methylsparteine under the influence of hydrogen iodide. These results confirm the analogy which exists between the two bases dimethylpiperidine and α -methylsparteine (compare Willstätter, *Abstr.*, 1900, i, 249; Valeur, this vol., i, 736).

E. H.

Iodine Derivatives of Strychnine and Brucine. JOZEF BURACZEWSKI and TAP. KOZŃIEWSKI (*Bull. Acad. Sci. Cracow*, 1908, 644—649).—By the addition of a somewhat concentrated solution of iodine in carbon disulphide to a hot alcoholic solution of strychnine or brucine, di-iodo-derivatives are precipitated, from which the alkaloids are regenerated by alcoholic alkalis; it is not yet determined whether the compounds are produced by addition or by substitution.

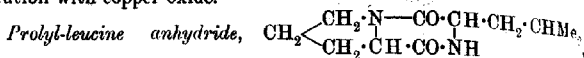
The *strychnine* derivative, $C_{21}H_{22}O_2N_2I_2$ or $C_{21}H_{20}O_2N_2I_2$, m. 196—197°, forms red crystals; after prolonged boiling with alcohol, the solution deposits ruby-red crystals of a substance, $C_{21}H_{22}O_2N_2I_2$, m. p. 253—254°, which seems to be identical with Jürgensen's periodide (*J. pr. Chem.*, 1870, [i], 2, 434). The *brucine* derivative, $C_{23}H_{26}O_4N_2I_2$ or $C_{23}H_{24}O_4N_2I_2$, m. p. 222.5°, is converted, after prolonged boiling with alcohol, into a ruby-red, crystalline substance, m. p. 251—252°. C. S.

A New Base from Putrefying Pancreas. D. ACKERMANN (*Zeitsch. physiol. Chem.*, 1908, 57, 28—29).—A new base, *viridinine*, $C_8H_{12}O_3N_2$, has been isolated from ox pancreas which had been allowed to undergo decomposition by exposure to the atmosphere during two months. It may be extracted by Kutscher's method, and is found in the same fraction as δ -aminovaleric acid, but may be separated from this by means of the slight solubility of its aurichloride or platinichloride. The *hydrochloride* forms glistening, green needles, only moderately soluble in cold water, and when heated produces yellow vapours with an aromatic odour. The *aurichloride*, $C_8H_{12}O_3N_2 \cdot H_2AuCl_4$, forms glistening, blackish-green or blackish-yellow plates, or, after recrystallisation, brownish-black needles, m. p. 176° (decomp.). The *platinichloride*, $(C_8H_{12}O_3N_2)_2 \cdot H_2PtCl_6$, has an intense yellow colour, and has m. p. 212—216° (decomp.). J. J. S.

Synthesis of Polypeptides. XXVII. 2. Derivatives of Proline. [Pyrrolidine-2-carboxylic Acid]. EMIL FISCHER and GEORG REIF (*Annalen*, 1908, 363, 118—135).—Levene and Beatty isolated from the product of the tryptic digestion of gelatin a prollyglycine anhydride which, on hydrolysis, yields glycine and optically active proline (Abstr., 1906, i, 718). As other similar substances may be formed by the decomposition of proteins, it was thought of interest to study the synthesis of polypeptides derived from active proline.

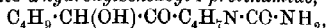
L-Proline, prepared from gelatin, is best purified by conversion into the *copper* salt, $C_{10}H_{16}O_4N_2Cu$, which crystallises from alcohol in dark blue, rhombic prisms, and when treated with hydrogen sulphide in hot aqueous solution yields proline, $[\alpha]_D^{20} - 76.7^\circ$. When treated with acetyl chloride, cooled by ice, and then with phosphorus pentachloride, *L*-proline forms *L*-prolyl chloride hydrochloride, $C_8H_9N \cdot COCl \cdot HCl$. This, when treated with glycine ethyl ester in chloroform solution at -10° , and then, after removal of the chloroform and excess of glycine ester, with dilute sodium ethoxide, yields prollyglycine anhydride and a brown oil. The anhydride, on extraction with boiling ethyl acetate or acetone, is obtained in microscopic leaflets, m. p. 213° (182—183°: Levene and Beatty, *loc. cit.*), $[\alpha]_D^{20} - 216.2^\circ$ to -217.4° , has a bitter

taste, and does not give a blue coloration when boiled in aqueous solution with copper oxide.



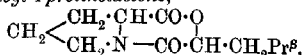
prepared in the same manner from *l*-prolyl chloride hydrochloride and *l*-leucine ethyl ester, crystallises from water in microscopic leaflets, m. p. 160° (corr.), $[\alpha]_D^{20} - 142.6^\circ$ to -143.4° . When boiled with 2*m*. hydrochloric acid, the anhydride yields proline, $[\alpha]_D^{20} - 56.9^\circ$, and leucine, $[\alpha]_D^{20} + 10.1^\circ$; hence racemisation must have taken place to the extent of one-third of the substance during the hydrolysis.

d- α -Bromoisohexoyl-*l*-proline, $\text{CHMe}_2 \cdot \text{CH}_2 \cdot \text{CHBr} \cdot \text{CO} \cdot \text{C}_4\text{H}_7\text{N} \cdot \text{CO}_2\text{H}$, formed by the action of *d*-bromoisohexoyl chloride and sodium hydroxide on *l*-proline, separates from acetone in crystals, m. p. 158° (corr.) (decomp.). When treated with concentrated aqueous ammonia at 25°, it is converted into α -hydroxyisohexoyl-*l*-prolinamide,



which is formed also by the action of liquid ammonia on bromoisohexoylproline. It crystallises from water in prisms, m. p. 123–124° (corr.), evolves ammonia at about 140°, $[\alpha]_D^{20} - 78.3^\circ$ to -78.6° , and yields ammonia when boiled with sodium hydroxide.

α -Hydroxyisohexoyl-*l*-prolinolactone,



formed when the amide is heated at 140–145°, or when *d*- α -bromoisohexoyl-*l*-proline is treated with sodium hydroxide at the ordinary temperature, and, after twenty-four hours, is neutralised with hydrochloric acid and evaporated, crystallises from water in thin needles, m. p. 164° (corr.), $[\alpha]_D^{20} - 166.8^\circ$, has a slightly bitter taste, and behaves towards alkalis and acids in the ordinary manner of the lactones.

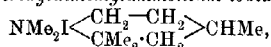
The substance described previously (Abstr., 1904, i, 917) as *i*-leucylproline is now found to be *i*-hydroxyisohexoyl prolinamide; on hydrolysis with acids, it yields ammonia, *dl*- α -hydroxyisohexic acid, and *dl*-proline. The anhydride, previously termed leucylproline anhydride (*loc. cit.*), is *i*-hydroxyisohexoylproline anhydride; it has m. p. 124° (corr.). G. Y.

New Isomerides of Coniine and other Hydrogenated Bases.
ICILIO GUARESCHI (*Atti R. Accad. Sci. Torino*, 1908, 43, 1095–1099. Compare following abstract).—No isomeride of coniine or other alkyl derivative of piperidine is known in which two alkyl groups are united to one and the same carbon atom. Such a derivative, namely, 2:2:4-trimethylpiperidine, should be obtainable from 5-cyano-2:2:4-trimethyldihydro-6-pyridone (compare Abstr., 1894, i, 484) by reduction with sodium and alcohol, whilst cyano-1-methylpyridones should yield the corresponding 1-methylpiperidines, 5-cyano-2:3:4-trimethyl-6-pyridone, and 2:3:4-trimethylpiperidine. In the last case, profound reduction by means of zinc dust gives 2:3:4-trimethylpyridine (compare Abstr., 1900, i, 558). Reduction of 3:5-dicyano-2:6-diketo-4-methyl-4-ethylhexahydropyridine should also give an isomeride of coniine, 4-methyl-4-ethylpiperidine, T. H. F.

New Isomeride of Coniine from 5-Cyano-2:2:4-trimethyldihydro-6-pyridone. GIOVANNI ISSOGGIO (*Atti R. Accad. Sci. Torino*, 1908, 43, 1100—1121. Compare preceding abstract).—The author has investigated the products of reduction of 5-cyano-2:2:4-trimethyldihydro-6-pyridone (compare Guareschi, *Abstr.*, 1893, i, 484) by means of (1) sodium and ethyl alcohol, and (2) sodium and amyl alcohol. The first method of reduction yields 2:2:4-trimethylpiperidine and 5-cyano-2:2:4-trimethylpiperidine (?) in small quantities, whilst the use of amyl alcohol leads to the formation of (1) 2:2:4-trimethylpiperidine, (2) a hexamethyldipiperidine, as the result of the condensing action of the excess of sodium on the preceding compound, and (3) 2:2:4-trimethylnipecotinic acid, derived from the complete hydrogenation of the pyridone and the hydrolysis of the cyanogen residue. The normal reduction of the cyanogen group to $\text{CH}_2\cdot\text{NH}_2$ by means of sodium and alcohol is not observed in this case.

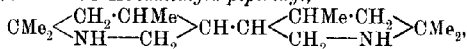
5-Cyano-2:2:4-trimethylpiperidine (?), $\text{NH} \begin{array}{c} \text{CH}_2\text{—CH(CN)} \\ \text{CMe}_2\text{—CH}_2 \end{array} \text{CHMe}$, crystallises from water in mammillary masses, m. p. 155°.

2:2:4-Trimethylpiperidine, $\text{NH} \begin{array}{c} \text{CH}_2\text{—CH}_2 \\ \text{CMe}_2\text{—CH}_2 \end{array} \text{CHMe}$, is a mobile liquid, b. p. 148°, D^{15}_4 0.832, has an intense alkaline reaction, and forms white fumes with hydrogen chloride; with copper sulphate it gives a blue precipitate, soluble in excess of reagent, and with Nessler's reagent, a white precipitate. The aqueous solution of its hydrochloride gives precipitates with the ordinary alkaloidal reagents. The *platinichloride*, $(\text{C}_8\text{H}_{17}\text{N})_2\cdot\text{H}_2\text{PtCl}_6$, forms orange-red, mammillary masses, m. p. 215—216° (decomp.). The *aurichloride*, $\text{C}_8\text{H}_{17}\text{N}\cdot\text{HAuCl}_4$, crystallises in yellow prisms, m. p. 135°. With methyl iodide, it gives 2:2:4-trimethylpiperidyltrimethylammonium iodide,

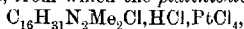


which separates from a mixture of alcohol and ether as a white, crystalline powder, m. p. 266° (decomp.).

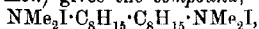
2:2:4:2':2':4'-Hexamethyldipiperidyl,



b. p. 266—267°/745 mm. and 237°/40 mm., D^{15}_4 0.869, has an odour resembling that of nicotine, and exhibits intense alkaline properties. Its *platinichloride*, $\text{C}_{16}\text{H}_{32}\text{N}_2\cdot\text{H}_2\text{PtCl}_6\cdot 3\text{H}_2\text{O}$, forms orange-yellow laminae, m. p. 262° (decomp.); its *picrate*, pale yellow needles, m. p. 230°; its *aurichloride*, $\text{C}_{16}\text{H}_{32}\text{N}_2\cdot 2\text{HAuCl}_4$, separates as a yellow powder, which changes gradually into laminae, m. p. 183—184°; its *mercurichloride*, $\text{C}_{16}\text{H}_{32}\text{N}_2\cdot 2\text{HgCl}_2$, forms short, colourless prisms, m. p. 270° (decomp.). When heated in a sealed tube with methyl iodide (2 mols.), the base (1 mol.) forms the compound, $\text{C}_8\text{H}_{16}\text{N}\cdot\text{C}_8\text{H}_{15}\cdot\text{NMe}_2\text{I}$, which is obtained as a dense oil, and, when evaporated with hydrochloric acid, gives a syrupy residue, from which the *platinichloride*,

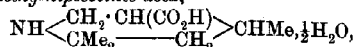


m. p. 214°, is obtained. With 4 mols. of methyl iodide in methyl alcohol, the base (1 mol.) gives the compound,



which crystallises from methyl alcohol in monoclinic laminae, m. p. 236—237°; the action of moist silver oxide gives the corresponding hydroxide, which is an energetic base, and in presence of hydrochloric acid gives the *platinichloride*, $C_{16}H_{30}N_2Me_4Cl_2 \cdot PtCl_4$, m. p. 227—228°.

2 : 2 : 4-*Trimethylnipecotinic acid*,



crystallises from alcohol in groups of hard, white prisms, m. p. 123° (efferves.).

T. H. P.

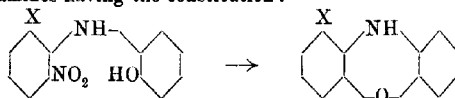
Preparation of Basic Compounds from Methyl Ethyl Ketone. WILHELM TRAUBE (D.R.-P. 200203).—Acetone absorbs much more ammonia than its homologues, giving rise to diacetoneamine, and in smaller proportion, triacetoneamine; but by saturating an alcoholic solution of methyl ethyl ketone, a basic condensation product, $C_{12}H_{23}ON$, may be obtained, having probably the constitution $NH \begin{array}{c} \diagup CM_2Et \cdot CHMe \\ \diagdown CM_2Et \end{array} \begin{array}{c} \diagdown CH_2 \\ \diagup \end{array} CO$. In accordance with this view of its structure, the compound is called either 2 : 3 : 6-trimethyl-2 : 6-diethyl-4-piperidone or trimethyldiethylketoneamine. It is a yellow oil, b. p. 247°/740 mm. and 157—159°/30 mm.; hydrochloride, m. p. 128°; sulphate, decomposes at 188°; nitrate, m. p. 169—172°; hydrogen oxalate, m. p. 183°. Another basic product, b. p. 104°/30 mm., is also formed in this condensation; and is separated from the preceding compound by means of its more soluble oxalate.

G. T. M.

Preparation of Anthracene Derivatives containing Nitrogen. FARBENFABRIKEN VORM. FRIEDR. BAYER & Co. (D.R.-P. 199713).—It was shown previously (compare this vol., i, 699) that the acetyl derivatives of the secondary α -aminoanthraquinones, when condensed with the aid of alkalis, gave rise to products of the pyridone series. Similarly, sulphonated pyridones are obtained from the sulphonic acids of acetyl- α -methylaminoanthraquinone.

G. T. M.

Preparation of Phenoxazines. ACTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKATION (D.R.-P. 200736).—The substituted nitrohydroxydiphenylamines having the constitution :



readily give rise to phenoxazines; this reaction, however, does not take place in the absence of a substituent in position 6, denoted by X.

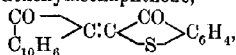
5-Nitrophenoxazine, reddish-violet needles, m. p. 165°, was obtained from 2 : 6-dinitro-2'-hydroxydiphenylamine by warming with 5% aqueous sodium hydroxide. The patent contains a description of six phenoxazine-carboxylic and -sulphonic acids derived from carboxy- and sulpho-nitro-2-hydroxy-diphenylamines.

G. T. M.

Some **Unsymmetrical Analogues of Indigotin**. ARMIN GROB (*Ber.*, 1908, 41, 3331—3334).—The two dyes referred to below have been recently described by Bezdrík and Friedländer (this vol., i, 674), but the author claims priority, as a patent for their preparation had been applied for by the Basler Chemische Fabrik in January—June, 1907.

8-Oxy-7-indoxylacenaphthene, $\text{C}_{10}\text{H}_6 \begin{array}{c} \text{CO} \diagup \\ \text{C} \diagdown \end{array} \text{C} \begin{array}{c} \text{CO} \diagdown \\ \text{NH} \diagup \end{array} \text{C}_6\text{H}_4$, gives an additive product with bromine, from which hydrogen bromide is expelled on heating, leaving a violet bromine-substituted dye. The solution of the leuco-compound of 8-oxy-7-indoxylacenaphthene is violet.

8-Oxy-7-oxythionaphthylacenaphthene,



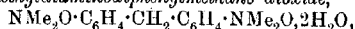
may be prepared by condensing *o*-carboxyphenylthioglycolic acid with acenaphthenequinone in acetic anhydride solution. It gives a bromine additive compound, and the solution of its leuco-compound is reddish-violet.

J. C. C.

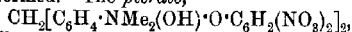
Amino-oxides of Leuco-bases of the Di- and Tri-phenylmethane Series. EUGEN BAMBERGER and LEO RUDOLF (*Ber.*, 1908, 41, 3290—3315).—The authors have investigated the action of hydrogen peroxide and of Caro's acid on tetramethyldiaminodiphenylmethane, tetramethyldiaminotriphenylmethane, and hexamethyltriminotriphenylmethane, and find that oxides are formed analogous to those obtained from dimethylaniline, etc., thus: $\text{R} \cdot \text{NMe}_2$.



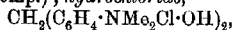
4:4'-Tetramethyldiaminodiphenylmethane dioxide,



is most conveniently prepared by the use of Caro's acid. It forms hard, colourless, glistening needles, m. p. 147°. When heated, it evolves vapours of formaldehyde. The aqueous solution has a faint alkaline reaction, and does not reduce Fehling's solution or oxidise potassium iodide. When exposed for three months over concentrated sulphuric acid at 12—15 mm. pressure, the anhydrous compound, m. p. 156°, is formed. The *picrate*,

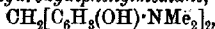


long, pale yellow needles, darkening at 130°, sintering at 145°, m. p. 150·5—151° (decomp.); *hydrochloride*,



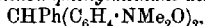
colourless, glistening needles, turning brown at 150°, m. p. 165·5—166° (decomp.); *platinichloride*, *aurichloride*, and *hydrogen-ferrocyanide* are described. On reduction, the hydrochloride yields tetramethyldiaminodiphenylmethane, and when iron is used, traces of tetramethyldiaminobenzhydrol and tetramethyldiaminobenzophenone appear to be formed. With nitrous acid, the hydrochloride gives 3:3'-dinitrotetramethyldiaminodiphenylmethane, and when the dioxide is treated with sulphur dioxide, *tetramethyldiaminodiphenylmethane-3:3'-disulphonic acid* is produced.

By treating the dioxide with acetic anhydride and sulphuric acid, the substance undergoes intramolecular change, partly into *tetramethyldiamino-3:3'-dihydroxydiphenylmethane*,



colourless, hard, glassy prisms, m. p. 114.5–115°, and partly, with simultaneous formation of *tetramethyldiaminodiphenylmethane*, into *tetramethyldiamino-3-hydroxydiphenylmethane*, white, silky needles, m. p. 111–111.5°.

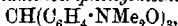
4:4'-*Tetramethyldiaminotriphenylmethane dioxide*,



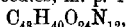
crystallises with $2\text{H}_2\text{O}$ (analysis by Rudolf) or $4\text{H}_2\text{O}$ (analysis by Reber), forming white, glistening needles, m. p. 131.5–132.5°. Its properties resemble those of the diphenylmethane derivative. The anhydrous substance has m. p. 188–189° (decomp.). The *picrate*, $\text{C}_{35}\text{H}_{32}\text{O}_{16}\text{N}_8$, *platinichloride*, and *hydrogen ferrocyanide* are described. On reduction, the dioxide yields leuco-malachite-green, and, when iron is used, also probably some *tetramethyldiaminotriphenylcarbinol*.

By the action of nitrous acid, an orange-yellow, uncrystallisable powder is formed, which is probably 3:3'-*dinitrotetramethyldiaminotriphenylmethane*, $\text{CHPh}[\text{C}_6\text{H}_3(\text{NO}_2)\cdot\text{NMe}_2]_2$; on reduction, this yields 3:3'-*diaminotetramethyldiaminotriphenylmethane*, pale brown, amorphous flakes; the *platinichloride*, $\text{C}_{23}\text{H}_{26}\text{N}_4\cdot\text{H}_2\text{PtCl}_6$, was prepared. By the action of acetic anhydride and sulphuric acid, the dioxide is converted into 4:4'-*tetramethyldiamino-3:3'-dihydroxytriphenylmethane*, colourless needles, m. p. 183–184°.

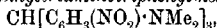
4:4':4''-*Hexamethyltriaminotriphenylmethane trioxide*,



forms hair-like, colourless needles, m. p. 176°; the *picrate*,



and *platinichloride* are described. On reduction, it yields leuco-crystal-violet, and by the action of nitrous acid it gives 3:3':3''-*trinitro-4:4':4''-hexamethyltriaminotriphenylmethane*,



orange-yellow flakes, which, on reduction, furnishes 3:3':3''-*triamino-4:4':4''-hexamethyltriaminotriphenylmethane*, white needles, m. p. 221.5–223.5°; the *platinichloride* is described.

By the action of acetic anhydride and sulphuric acid, the trioxide is converted into leuco-crystal-violet and 4:4':4''-*hexamethyltriamino-3:3':3''-trihydroxytriphenylmethane*, a pale bluish-green powder.

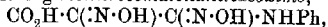
J. C. C.

Negative Substituted Amino-oximes. II. Phenylnitroethenylamino-oxime. WILHELM STEINKOPF and CZESLAU BENEDEK (*Ber.*, 1908, 41, 3566–3568. Compare Steinkopf and Bohrmann, *Abstr.*, 1907, i, 480; this vol., i, 327).—It has been shown previously that the ease with which the halogenated and nitro-substituted ethenylamino-oximes are formed, and their comparative stability towards water, results from the negative influence of the substituting groups. It was of interest, therefore, to study the influence of a phenyl-substituting group, which is negative like the

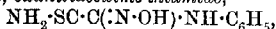
halogens and nitro-group, but occupies a greater volume. It is now found that, whilst phenylnitroethenylamino-oxime is readily formed, it is unstable, and decomposes in a closed vessel in the course of a few days, or more slowly in an open vessel. Hydroxylamine can be detected in the cold aqueous solution of the amino-oxime.

Phenylnitroethenylamino-oxime, $\text{NO}_2\cdot\text{CHPh}\cdot\text{C}(\text{NOH})\cdot\text{NH}_2$, prepared by the action of hydroxylamine hydrochloride on a concentrated aqueous solution of sodium *aci*-phenylnitroacetonitrile at the laboratory temperature, forms white crystals, m. p. 125° , reduces mercuric chloride in cold alkaline solution, gives a violet coloration with ferric chloride, and forms a *hydrochloride*, which separates in white crystals, m. p. 153° . The amino-oxime acts as a dibasic acid, forming two series of salts. The *copper*, $\text{C}_8\text{H}_7\text{O}_3\text{N}_3\text{Cu}$, and *barium*, $(\text{C}_8\text{H}_7\text{O}_3\text{N}_3)_2\text{Ba}$, salts were analysed. G. Y.

Indigotin Group. HEINRICH WIELAND and ERWIN GMELIN (*Ber.*, 1908, 41, 3512—3517. Compare Wieland and Semper, this vol., i, 108).—The ethyl glyoximeperoxidedicarboxylate discovered by Präpper (*Annalen*, 1884, 222, 46) is converted by barium hydroxide into barium furoxandicarboxylate. This salt reacts with aniline hydrochloride, forming *isonitrosomalonanilideoxime*,



and this when warmed with acetic anhydride is converted into *cyano-N-acetylformanilideoxime*, $\text{CN}\cdot\text{C}(\text{N}\cdot\text{OH})\cdot\text{NAcPh}$. On warming with ammonium sulphide, *oxanilideoxime-thiamide*,



is formed, which, on warming with concentrated sulphuric acid, gives *α*-isatoxime, $\text{C}_6\text{H}_4\cdot\text{C}(\text{CO})\text{NH}\cdot\text{C}\cdot\text{N}\cdot\text{OH}$.

Ethyl furoxandicarboxylate is hydrolysed with baryta and heated at $30\text{--}40^\circ$ with concentrated aqueous aniline hydrochloride. The *aniline* salt of *isonitrosomalonanilideoxime* separates in yellow crystals, m. p. 133° ; the corresponding acid obtained by hydrolysis with hydrogen chloride forms colourless plates, m. p. $165\text{--}166^\circ$ (decomp.); it gives a blackish-green coloration with ferric chloride. The *barium* salt is light yellow; the *sodium hydrogen* salt, colourless. *Cyano-N-acetylformanilideoxime* crystallises in large, colourless prisms, m. p. 135° ; it does not give a coloration with ferric chloride. *Oxanilideoxime-thiamide* forms bright yellow crystals, m. p. $169\text{--}171^\circ$, and shows the characteristic dark green ferric chloride reaction. It is added in portions to sulphuric acid, maintained at 60° , and the temperature is gradually raised until sulphur dioxide fumes are evolved, when the solution is poured on to ice, neutralised, and the *α*-isatoxime extracted with ethyl acetate. E. F. A.

Hydrazones of Sugars. A. RECLAIRE (*Ber.*, 1908, 41, 3665—3671).—The *o*-, *m*-, and *p*-nitrophenylhydrazones of various sugars have been prepared in aqueous-alcoholic or in acetic acid solution. Many of them have already been described by Alberda van Ekenstein and

Blanksma (Abstr., 1904, i, 98). The following new compounds are mentioned: Dextrose forms a *m*-nitrophenylhydrazone, m. p. 115—116°, *m*-nitrophenylosazone, m. p. 228°, *o*-nitrophenylhydrazone, m. p. 148°, and *o*-nitrophenylosazone, m. p. 215—217°. Mannose forms a *m*-nitrophenylhydrazone, m. p. 162—163°, an impure *m*-nitrophenylosazone, m. p. 214° (decomp.), and an *o*-nitrophenylhydrazone, m. p. 173°. Lævulose forms an *o*-nitrophenylhydrazone, m. p. 155—156°. Galactose forms a *m*-nitrophenylhydrazone, m. p. 181—182°, and an *o*-nitrophenylhydrazone, m. p. 172. Arabinose forms a *m*- and an *o*-nitrophenylhydrazone, m. p. 179—180° and 180° respectively. Xylose forms a *m*-nitrophenylhydrazone, m. p. 130° (decomp. at 120°). Rhamnose forms a *m*- and an *o*-nitrophenylhydrazone, m. p. 104—105° and 151° respectively. C. S.

Action of Secondary *as*-Hydrazines on Carbamide. HUGO MILRATH (*Monatsh.*, 1908, 29, 909—935).—Most of the matter in this communication has already been abstracted (this vol., i, 581). Phenylbenzylsemicarbazide, when heated with acetic anhydride, yields *diacetyl-as-phenylbenzylhydrazine*, $C_{17}H_{18}O_2N_2$, m. p. 128° (compare Milrath, *loc. cit.*), which may also be prepared directly from *as*-phenylbenzylhydrazine.

Diphenyldimethylsemicarbazide may be prepared by heating carbamide (1 mol.) with *as*-phenylmethylhydrazine (2 mols.) at 170—180°.

The substance, m. p. 167—168°, described previously (*loc. cit.*), is now shown to be 1-benzylbenzopyrazolone, $C_6H_4 \begin{smallmatrix} \text{N}(\text{CH}_2\text{Ph}) \\ \text{CO} \end{smallmatrix} \text{NH}$.

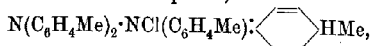
It may also be prepared by acting on benzopyrazolone (anhydro-*o*-hydrazinobenzoic acid: Fischer and Renouf, Abstr., 1882, 1068) with benzyl chloride and sodium methoxide. The white, crystalline hydrochloride, $C_{14}H_{12}ON_2 \cdot HCl$, yellow, crystalline *platinichloride*, m. p. 134—138° (decomp.), and *picrate*, $C_{14}H_{12}ON_2 \cdot C_6H_3O_7N_3$, large, dark yellow plates, m. p. 154—155° (decomp.), were prepared. 1-Benzylbenzopyrazolone in alkaline solution is converted by methyl sulphate into 1-benzyl-2-methylbenzopyrazolone, $C_6H_4 \begin{smallmatrix} \text{N}(\text{CH}_2\text{Ph}) \\ \text{CO} \end{smallmatrix} \text{NMe}$, which crystallises with $1H_2O$ in long, slender, silky, white needles, softens at 65°, and has m. p. 75—80°.

1-Methylbenzopyrazolone, $C_6H_4 \begin{smallmatrix} \text{NMe} \\ \text{CO} \end{smallmatrix} \text{NH}$, obtained by heating carbamide (1 mol.) with *as*-phenylmethylhydrazine at 170—180°, and finally at 270—280°, has m. p. 153—154°. W. H. G.

Decomposition of Tetra-arylhydrazines. IV. HEINRICH WIELAND (*Ber.*, 1908, 41, 3478—3498. Compare Abstr., 1907, i, 1076).—Aromatic hydrazines are decomposed by various acid reagents in accordance with the equation: $\text{Ar}_2\text{N} \cdot \text{NAr}_2 + \text{HR} \rightarrow \text{Ar}_2\text{NH} + \text{RNR}_2$. Diarylamines and a molecule comprising the acid residue R result. In the case of hydrogen chloride, the hypothetical product will be a diarylamine containing chlorine attached to nitrogen, NAr_2Cl . Such secondary aromatic chloramines are not known, and attempts to

synthesise them have only yielded hydrazines, $\text{Ar}_2\text{N}\cdot\text{NAr}_2$, or compounds substituted in the nucleus. On decomposition of tetraphenylhydrazine, two molecules of chloroamine unite, with elimination of hydrogen chloride, in the para-position, $\text{C}_6\text{H}_5\cdot\text{NCl}\cdot\text{C}_6\text{H}_4\cdot\text{NPh}_2$. This primary product undergoes rearrangement to *p*-chloroanilinetriphenylamine, $\text{Cl}\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{NPh}_2$ (Gambarjan, following abstract).

In the case of tetra-*p*-tolylhydrazine, two molecules of the chloroamine unite in the ortho-position, two mols. of hydrogen chloride being eliminated, and a completely substituted phenazine ring compound is obtained. In addition, chlorinated phenazine compounds containing two halogen atoms in the nucleus are formed, due to the oxidising action of the chloroamine. The first product of the decomposition is the violet quinonoid additive compound,



already described (Abstr., 1907, i, 1076).

Other acids act similarly to hydrogen chloride, and the primary products of the type $\text{NAr}_2\cdot\text{OR}$ are to be regarded as derivatives of the hypothetical diphenylhydroxylamine, $\text{NPh}_2\cdot\text{OH}$.

Tetra-anisylhydrazine is not obtained on oxidation of *p*-dianisylamine, but a ditertiary azine is formed by elimination of water from two molecules of the unstable hydroxylamine.

The ditertiary phenazine derivatives, conveniently termed "perazines," are stable towards oxidising agents. They form coloured salts with mineral acids, carboxylic acids, or phenols in anhydrous solvents, which are, as a rule, mono-acid, and are regarded as quaternary quinolimonium compounds. On oxidation, a series of salts of other colours are obtained, which possibly represent the *o*-quinonoidbis-phenazonium series.

By the action of hydrogen chloride in ether on tetratolylhydrazine in chloroform solution, the violet solution of the quinolimonium salt is first obtained, which changes subsequently to red, and deposits glistening, dark green, flat needles of *dichloroditolyltolazonium hydrochloride*. Alkalis decompose this into *dichloroditolylidihydrotolazine*, which is obtained from xylene in lustrous, garnet-red, four-sided prisms. It is very stable, and begins to decompose above 360° . With bromine, it forms a dark violet additive product containing one mol. of bromine. By reduction with sodium and amyl alcohol, *p*-ditolylidihydrotolazine, $\text{C}_6\text{H}_3\text{Me}\langle\text{N}(\text{C}_6\text{H}_4\text{Me})\rangle\text{C}_6\text{H}_3\text{Me}$, is quantitatively formed, crystallising in long, orange-yellow, glistening needles, m. p. 269° ; it can be distilled without decomposition. Both ditolylidihydrotolazine and its dichloro-derivative are formed by the action of hydrogen chloride on tetratolylhydrazine, but the latter is the main product.

Ditolylidihydrotolazine itself does not exhibit the properties of a base, and can be heated with aqueous acids without forming salts. With alcoholic or ethereal acid solutions, it forms dark green azonium salts, of which the *hydrochloride* is particularly characteristic; it separates in dark green, glistening, broad needles. The normal salt contains one molecule of acid, but there is a tendency to form an acid

salt. The *nitrate* forms minute, dark green needles, decomp. 256°. Decomposition of the salts with sodium hydroxide leads to the formation of basic by-products; alcoholic potassium hydroxide or zinc dust afford better means of effecting the decomposition.

Acetic acid acts very readily on tetratolylhydrazine; the solution becomes at first dark violet, owing to the formation of the quinonoid hydrazoneium acetate, and then dark green, owing to conversion into tolazonium acetate. On adding water, *p*-ditolylamine separates, and dityldihydrotolazine may be obtained from the mother liquors.

Phosphoryl chloride or phosphorus trichloride and benzoyl chloride do not react with tetratolylhydrazine in the absence of water. When moisture is present, large, dark green crystals are obtained; their formation is due to the hydrogen chloride slowly liberated by the chlorides, which gives rise to dichlorotolazonium chloride and phosphate.

p-*Dianisylamine*, $(\text{OMe}\cdot\text{C}_6\text{H}_4)_2\text{NH}$, prepared by Goldberg's method (this vol., i, 17), does not yield tetra-anisylhydrazine on oxidation, but an *azine*, $\text{C}_{26}\text{H}_{26}\text{O}_4\text{N}_2$, crystallising in stellar aggregates of bright, yellow needles, m. p. 290°. The *anisazonium* salts are similar to the tolazonium compounds; the *hydrochloride* forms green, glistening needles; the *nitrate* is obtained as a wine-red precipitate, m. p. 223° (decomp.). The *ferrichloride* separates in bronze, glistening, violet-red plates; the *chromate* forms dark red needles; the *platinichloride* is insoluble in water, and likewise the *ferro*- and *ferri-cyanides*, of which the former yields a rose-red, the latter a brownish-red, precipitate.

Wool is quickly dyed by anisazonium salts in fast, scarlet-red shades. Anisazonium chloride is dissolved by pyridine without change, but gives an intense sea-blue coloration with aniline. Oxidising agents convert the anisazonium salts into green compounds, which are unstable, and pass slowly in solution into carmine substances. The new perazonium salts have characteristic absorption bands in their spectra.

E. F. A.

Decomposition of Tetraphenylhydrazine with Hydrogen Chloride. STEFAN GAMBARJAN (*Ber.*, 1908, 41, 3507--3512).—In a previous paper (Wieland and Gambarjan, *Abstr.*, 1906, i, 453) it was stated that tetraphenylhydrazine, when treated with hydrogen chloride in anhydrous ethereal solution, formed diphenylamine hydrochloride and *p*-chlorodiphenylamine. It is now found that the latter compound is not produced, but that a secondary base, *p*-chloroanilino-triphenylamine, is formed. The constitution of this has been established by elimination of the chlorine by means of sodium and the identification of the product with *anilino-triphenylamine* (triphenyl-*p*-phenylenediamine), prepared synthetically by treating diphenylamine with *p*-iodonitrobenzene, reducing the nitro group, acetylating, and again treating with iodobenzene.

The decomposition of the tetraphenylhydrazine is quantitative. The dark green quinolimonium salt is first formed, and decomposes

to colourless diphenylamine and diphenylchloroamine. Two molecules of the latter then interact, and chlorine is eliminated.

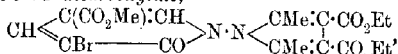
p-Chloroanilinotriphenylamine, $\text{NPh}_3 \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdot \text{C}_6\text{H}_4\text{Cl}$, has b. p. $205-215^\circ/0.01$ mm., m. p. $77-81^\circ$, and forms a crystalline hydrochloride and an acetyl derivative, m. p. $199-200^\circ$. *p*-Anilinotriphenylamine, prepared from this by means of sodium, has m. p. 134° , and yields an acetyl derivative, m. p. 184° .

p-Nitrotriphenylamine crystallises in rectangular plates, m. p. 144° , insoluble in acetic acid. *p*-Aminotriphenylamine has m. p. $145-148^\circ$, and forms an acetyl derivative, crystallising in silvery plates, m. p. 195° .

E. F. A.

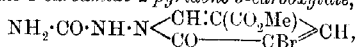
Action of *N*-Amino-compounds on Bromocoumalinic Acid Esters. CARL BÜLOW and HANS FILCHNER (*Ber.*, 1908, **41**, 3281—3285).—Compounds containing two similar ring systems united by the $>\text{N} \cdot \text{N} <$ group have been described by Bülow and Sautermeister (*Abstr.*, 1904, i, 690) and by von Pechmann and Mills (*Abstr.*, 1904, i, 1042). Bülow (*Abstr.*, 1906, i, 905; 1907, i, 99) has also prepared a compound containing two different five-membered rings united by the same grouping. The authors have not succeeded in preparing a compound containing a five-membered and a six-membered ring joined together by the group $>\text{N} \cdot \text{N} <$.

5-Methyl 3':4'-diethyl 1'-(3-bromo-5-carboxy-2-pyridonyl)-2':5'-dimethylpyrrole-3':4'-dicarboxylate,



prepared by condensing methyl bromocoumalinate with ethyl 1-amino-2:5-dimethylpyrrole-3:4-dicarboxylate in presence of glacial acetic acid, forms colourless needles, m. p. 155° .

Methyl 3-bromo-1-carbamido-2-pyridone-5-carboxylate,



prepared by condensing semicarbazide with methyl bromocoumalinate, forms colourless needles, m. p. 223° . On hydrolysis, it yields 3-bromo-1-carbamido-2-pyridone-5-carboxylic acid, m. p. 252° . J. C. C.

Preparation of 5:5-Dialkylbarbituric Acids. CHEMISCHE FABRIK AUF ACTIEN (VORM. E. SCHERING) (*D.R.P.* 201244).—Dimethyl- and diethyl-malonylguanidines, when hydrolysed with boiling 10% sulphuric acid, or at 120° with concentrated hydrochloric or hydrobromic acid, give rise to the corresponding 5:5-dialkylbarbituric acids. G. T. M.

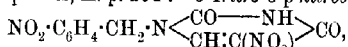
4:6-Dihydroxy-2-phenylpyrimidine. E. L. PINNER (*Ber.*, 1908, **41**, 3317—3519).—4:6-Dihydroxy-2-phenylpyrimidine, $\text{C}_{10}\text{H}_8\text{O}_2\text{N}_2$, produced by the interaction of equivalent quantities of benzamidine hydrochloride and ethyl malonate in the presence of sodium ethoxide at the ordinary temperature, forms crystals, m. p. $325-330^\circ$. It is converted by bromine in glacial acetic acid into 5-bromo-4:6-dihydroxy-2-phenylpyrimidine, $\text{C}_{10}\text{H}_7\text{O}_2\text{N}_2\text{Br}$, crystallising in pale yellow needles, m. p. 320° (decomp.), and by acetic anhydride into a monoacetate,

$C_{12}H_{10}O_2N_2$, glistening, flat prisms, m. p. 195° , and a *diacetate*, $C_{14}H_{12}O_4N_2$, brilliant, flat prisms, m. p. $93-94^\circ$. 4-Hydroxy-6-ethoxy-2-phenylpyrimidine, $C_{12}H_{12}O_3N_2$, is formed by heating the dihydroxy-compound with ethyl bromide and an alcoholic solution of potassium hydroxide under pressure at 100° ; it crystallises in flat prisms, m. p. 174° .

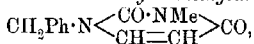
W. H. G.

Pyrimidines. XXXVIII. Syntheses of some Benzyl Derivatives of Uracil and Thymine. TREAT B. JOHNSON and JOHN H. DERBY, jun. (*Amer. Chem. J.*, 1908, 40, 444-458).—The authors have synthesised 1- and 3-benzyluracil and 1- and 3-benzylthymine.

2-Ethylthiol-6-oxy-1-benzylpyrimidine, $N \begin{smallmatrix} \text{C(SET) \cdot N(CH}_2\text{Ph)} \\ \text{CH} \text{---} \text{CH} \end{smallmatrix} \text{CO}$, prepared by the action of benzyl chloride on 2-ethylthiol-6-oxy-pyrimidine, forms prisms, m. p. 77° . 2-Ethylthiol-6-oxy-3-benzylpyrimidine, $CH_2Ph \cdot N \begin{smallmatrix} \text{C(SET) \cdot N} \\ \text{CH} \text{---} \text{CH} \end{smallmatrix} \text{CO}$, formed by the same reaction and isolated through its insolubility in ether, crystallises in long slender prisms and hexagonal prisms or flat tablets, m. p. 136° . 5-Bromo-2-ethylthiol-6-oxy-3-benzylpyrimidine, prepared by the interaction of benzyl chloride and 5-bromo-2-ethylthiol-6-oxy-pyrimidine (Wheeler and Johnson, *Abstr.*, 1904, i, 624), crystallises in needles, m. p. 129° . 1-Benzyluracil, $NH \begin{smallmatrix} \text{CO \cdot N(CH}_2\text{Ph)} \\ \text{CH} \text{---} \text{CH} \end{smallmatrix} \text{CO}$, prepared by evaporating 2-ethylthiol-6-oxy-1-benzylpyrimidine with hydrochloric acid, forms prismatic crystals, m. p. 175° . It gives a red colour with diazobenzenesulphonic acid. 3-Benzyluracil, prepared similarly from 2-ethylthiol-6-oxy-3-benzylpyrimidine, crystallises in stout prisms, m. p. 173° . It gives only a yellow colour with diazobenzenesulphonic acid (distinction from 1-benzyluracil). 5-Bromo-3-benzyluracil, prepared by brominating 3-benzyluracil or by digesting 5-bromo-2-ethylthiol-6-oxy-3-benzylpyrimidine with concentrated hydrochloric acid, forms hexagonal prisms, m. p. 204° . 5-Nitro-3-p-nitrobenzyluracil,



prepared by nitrating 3-benzyluracil, crystallises in slender prisms, decomposing at $235-240^\circ$. 3-Benzyl-1-methyluracil,



prepared by treating 3-benzyluracil with methyl iodide or from 1-methyluracil and benzyl chloride, forms needles, m. p. 75° . On bromination, it gives 5-bromo-3-benzyl-1-methyluracil, which is also formed by the interaction of methyl iodide and 5-bromo-3-benzyluracil; it crystallises in diamond-shaped prisms, m. p. 123° . 2-Ethyl-

thiol-6-oxy-1-benzyl-5-methylpyrimidine, $N \begin{smallmatrix} \text{C(SET) \cdot N(CH}_2\text{Ph)} \\ \text{CH} \text{---} \text{CMe} \end{smallmatrix} \text{CO}$,

prepared by the interaction of benzyl chloride and 2-ethylthiol-6-oxy-5-methylpyrimidine, forms stout prisms, m. p. 70° ; in the same reaction there is also formed (insoluble in ether) 2-ethylthiol-6-oxy-3-benzyl-5-methylpyrimidine, crystallising in hexagonal plates, m. p.

21—122°. 1-Benzylthymine, $\text{NH} \begin{array}{c} \text{CO} \cdot \text{N}(\text{CH}_2\text{Ph}) \\ \text{CH} = \text{CMe} \end{array} \text{CO}$, prepared by digesting 2-ethylthiol-6-oxy-1-benzyl-5-methylpyrimidine with concentrated hydrochloric acid, forms clusters of radiating prisms, m. p. 204—205°; with diazobenzenesulphonic acid it gives a claret-red solution. 3-Benzylthymine, $\text{CH}_2\text{Ph} \cdot \text{N} \begin{array}{c} \text{CO} \cdot \text{NH} \\ \text{CH} : \text{CMe} \end{array} \text{CO}$, similarly prepared from 2-ethylthiol-6-oxy-3-benzyl-5-methylpyrimidine, forms diamond-shaped prisms, m. p. 160°. It gives only a yellow colour with diazobenzenesulphonic acid. 3-Benzyl-1-methylthymine, obtained by the action of methyl iodide on 3-benzylthymine, forms prismatic crystals, m. p. 101°; it does not react with diazobenzenesulphonic acid. On nitration it yields 5-nitro-4-hydroxy-3-p-nitrobenzyl-dihydrothymine, $\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH}_2 \cdot \text{N} \begin{array}{c} \text{CO} \text{---} \text{NH} \\ \text{CH}(\text{OH}) \cdot \text{CMe}(\text{NO}_2) \end{array} \text{CO}$, which forms prisms decomposing at 176°.

J. C. C.

Preparation of 4-Dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone. EMIL SCHEITIN (D.R.-P. 199844).—4-Dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone is readily obtained in good yield by heating 4-sulphonylamino-1-phenyl-2:3-dimethyl-5-pyrazolone with methyl sulphate in alcoholic solution at 100—115° (compare this vol., i, 688).

G. T. M.

Preparation of Chlorobromindigotin. GESELLSCHAFT FÜR CHEMISCHE INDUSTRIE IN BASEL (D.R.-P. 198816. Compare this vol., i, 695).—Chlorobromindigotin, obtained by heating at 226° in nitrobenzene, chloroindigotin (1 mol.) and bromine (1 mol.), is a blue, crystalline powder, sparingly soluble in water, alcohol, or benzene, but dissolving more readily in warm aniline or nitrobenzene.

On reduction with alkaline reducing agents, it furnishes a light yellow leuco-derivative.

G. T. M.

Halogen Indigotins. CARL G. SCHWALBE and HERMANN JOCHHEIM (*Ber.*, 1908, 41, 3796—3798).—A table is given of the colours of *o*-, *m*-, and *p*-dichloroindigotin and indigotin in sulphuric acid, alcohol, chloroform, acetic acid, pyridine, aniline, and nitrobenzene solutions, together with observations on the preparation of the three chloro-*o*-nitrobenzaldehydes; Müller's fourth isomeride is a mixture of *m*-chloro-*o*-nitrobenzaldehyde and unchanged aldehyde. W. R.

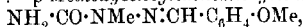
Halogen Indigotinsulphonic Acids. CARL G. SCHWALBE and HERMANN JOCHHEIM (*Ber.*, 1908, 41, 3798—3802).—A halogen phenylglycine mixed with sand, when washed with excess of cold sulphuric acid (60% SO_3), yields a halogen indigotinsulphonic acid; the purification is carried out by extraction with amyl alcohol and fractional precipitation with petroleum or benzene, the usual methods not giving satisfactory results. *m*-Chlorophenylglycine gives a tetrachlorotetrasulphonic acid; the *p*-chloro-derivative, a dichlorohexasulphonic acid, and the 2:4-dichloro-compound, a tetrachlorodisulphonic acid; *o*-chlorophenylglycine does not form a dye; the *o*-bromo-

compound gives a dye which does not contain halogen. A comparison of the aqueous solutions of these sulphonic acids and indigo-carmin as regards colour and dyeing properties is given. W. R.

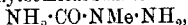
Constitution of the Acid Derivatives of Methylhydrazine.
AUGUST MICHAELIS and ERICH HADANCK (*Ber.*, 1908, **41**, 3285—3296).

—In attempting to prepare 5-phenyl-1-methyl-3-pyrazolone by the action of phosphorus trichloride on a mixture of ethyl benzoylacetate and an acid derivative of methylhydrazine, the authors invariably obtained 3-phenyl-1-methyl-5-pyrazolone, which is also produced by the condensation of methylhydrazine and ethyl benzoylacetate. As 3-pyrazolones are so often obtained by Michaelis and Mayer's reaction, the above fact could only be explained by the migration of the methyl group from the one nitrogen atom to the other, or else by the more probable assumption that the acid derivatives of methylhydrazine employed have an unsymmetrical constitution. The authors find that methylsemicarbazide, methylthiosemicarbazide, and mono- and dibenzoylmethylhydrazines have an unsymmetrical constitution, as they condense readily with aldehydes with the formation of hydrazones, whilst oxalyldimethylhydrazine appears to be symmetrically constituted.

Benzylidenemethylsemicarbazide, $\text{NH}_2\cdot\text{CO}\cdot\text{NMe}\cdot\text{N}:\text{CHPh}$, prepared by the action of benzaldehyde on methylsemicarbazide, forms white needles, m. p. 162° . *p-Methoxybenzylidenemethylsemicarbazide*,



similarly prepared from anisaldehyde, forms silky needles, m. p. 191° . The constitution of methylsemicarbazide is therefore



and, in accordance with this, it reacts with carbon disulphide to form *bis-methylcarbamidothiocarbamide*, $\text{CS}(\text{NH}\cdot\text{NMe}\cdot\text{CO}\cdot\text{NH}_2)_2$, white needles, m. p. $253\cdot5^\circ$. When methylhydrazine is treated with carbon disulphide, there is formed the *salt*, $\text{NHMe}\cdot\text{NH}\cdot\text{CS}\cdot\text{S}\cdot\text{NH}_2\cdot\text{NHMe}$, which loses hydrogen sulphide and yields *bis-methylaminothiocarbamide*, $\text{CS}(\text{NH}\cdot\text{NHMe})_2$, white needles, m. p. 241° .

Phenylbenzylidenemethylthiosemicarbazide, $\text{NHPh}\cdot\text{CS}\cdot\text{NMe}\cdot\text{N}:\text{CHPh}$, prepared by the interaction of benzaldehyde and phenylmethylthiosemicarbazide, forms long, glistening needles, m. p. $131\cdot5^\circ$.

Benzoylbenzylidenemethylhydrazine, $\text{NMeBz}\cdot\text{N}:\text{CHPh}$, prepared by the interaction of benzaldehyde and crude *monobenzoylmethylhydrazine* (an oil from benzoic anhydride and methylhydrazine), forms small, white needles, m. p. 82° .

Benzoylanisylidenemethylhydrazine, $\text{NMeBz}\cdot\text{N}:\text{CH}\cdot\text{C}_6\text{H}_4\cdot\text{OMe}$, prepared from anisaldehyde, forms slender, white needles, m. p. 115° . When benzoylmethylhydrazine in alcoholic or ethereal solution is shaken with yellow mercuric oxide, an oil is obtained which has the properties of a tetrazone; this could not, however, be prepared in the pure state.

Dibenzoylmethylhydrazine can be prepared by treating methylhydrazine with benzoic anhydride; it has m. p. 145° (von Brünig, *Abstr.*, 1890, 23, gives 143°).

Diacetyl-methylhydrazine, $\text{NMeAc} \cdot \text{NHAc}$, is a colourless oil, b. p. 280° .

Oxalyl-dimethylhydrazine does not yield a hydrazone with either benzaldehyde or anisaldehyde. J. C. C.

Constitution of the Endoxypyrrrodiazoles [4:5-Oxy-1:2:5-osotriazoles]. GIACOMO PONZIO (*Gazzetta*, 1908, 38, ii, 522-524. Compare Abstr., 1898, i, 386; 1899, i, 717, 827; 1900, i, 588; 1901, i, 169; 1902, i, 190).—The author's conclusion, that the compounds obtained by the oxidation of the hydrazoximes (*loc. cit.*) contain the nucleus $\text{O} \begin{array}{c} \text{N} \cdot \text{N} \\ \diagup \quad \diagdown \\ \text{C} \quad \text{C} \end{array} \text{N}$, is confirmed by the recent work of Wieland

and Semper (this vol., i, 108), who find that phosphorus pentachloride does not react with compounds containing the group $\text{N}:\text{O}$, whilst it readily transforms compounds containing the complex $\text{O} \begin{array}{c} \text{C} \\ \diagup \quad \diagdown \\ \text{N} \end{array}$ into the corresponding compounds containing the grouping :C:N .

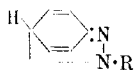
The interaction of phosphorus pentachloride and 4:5-oxy-1-phenyl-3:4-dimethyl-1:2:5-osotriazole yields 1-phenyl-3:4-dimethyl-1:2:5-osotriazole, which partly undergoes chlorination to 1-*p*-chlorophenyl-3:4-dimethyl-1:2:5-osotriazole. T. H. P.

Cain's Theory of Diazo-compounds and Ammonium Salts.

ARTHUR HANTZSCH (*Ber.*, 1908, 41, 3532-3536).—A criticism of Cain's theory of diazo-compounds (compare *Trans.*, 1907, 91, 1049) and of ammonium salts. The main objections raised by the author are briefly as follows: (1) diazobenzene salts have the properties of true benzene derivatives, and not those of derivatives of dihydro-



benzene, as represented by the annexed formula; (2) the behaviour of diazo-salts when reduced or oxidised, or when treated with halogen, is not in agreement with the presence of an unsaturated ring, as shown by this formula; (3) a compound having such a constitution, when reduced, should yield *p*-phenylenediamine; (4) since both normal and isodiazo-compounds are very similar in their chemical behaviour,



they cannot be represented by formulæ so different as those annexed. The fact that the isomeric diazocyanides give similar absorption spectra (compare Dobbie and Tinkler, *Trans.*, 1905, 87, 273) shows that these compounds must have very similar constitutions; (5) if tetraethylammonium chloride has the formula $\text{NEt}_4 \cdot \text{Cl} \cdot \text{Et}$, and undergoes ionisation in aqueous solution, thus: $\text{NEt}_3 \cdot \text{Cl} \cdot \text{Et} \rightarrow \text{NEt}_3 + \text{Et} \cdot \text{Cl} \rightarrow \text{NEt}_4^+ + \text{Cl}^-$, it should be possible to detect the triethylamine and ethyl chloride produced as intermediate products, since these compounds interact but very slowly at low temperatures. As the formation of these compounds cannot be detected, the above supposition is untenable. W. H. G.

Decomposition of Diazo-solutions. ARTHUR HANTZSCH and KENWORTHY J. THOMPSON (*Ber.*, 1908, 41, 3519-2532).—An

investigation on the rates of decomposition of aqueous solutions of various diazo-salts, carried out with the object of clearing up several points which were still unsettled (compare Schwalbe, Abstr., 1905, i, 618; Cain, *ibid.*, i, 724).

Freshly prepared diazobenzene chloride contains traces of a substance, the nature of which could not be determined, which acts as a preservative. It disappears when the diazo-compound is kept for some time in a desiccator, or when a current of dry air is passed over the substance; consequently, solutions of the freshly prepared salt are more stable ($k=0.0008-0.0009$) than those of the substance which has been kept for some days, or treated with a current of dry air, when the maximum value of k found was 0.00118 (compare Hantzsch and Osswald, Abstr., 1900, i, 703; Euler, Abstr., 1903, i, 298). It is therefore evident that the keeping qualities of aqueous solutions of diazo salts may be greatly influenced by very slight traces of unknown impurities. Diazobenzene bromide in dilute solutions decomposes at about the same rate as the chloride. The velocity of decomposition increases slowly with the concentration, the increase in the case of the bromide being slightly greater than that of the chloride, probably because the undissociated bromide is less stable than the undissociated chloride. Further, the velocity of decomposition increases with the decomposition of the salt; this is probably due to the formation of a halogenated benzene, produced by the action of the hydrogen halide which is liberated. Diazobenzene iodide, even in very dilute solutions, decomposes far more rapidly than the chloride or bromide, probably because the chief reaction which takes place in this case, namely, the formation of iodobenzene, proceeds at a greater rate than the production of phenol. A velocity constant could not be obtained, owing to the complexity of the many reactions which take place during the decomposition of solutions of diazonium iodides.

The rate of decomposition of *p*-nitrodiazobenzene chloride increases with the concentration; since it is greatly accelerated by light, the solutions were kept as far as possible in the dark, the value obtained being $k=0.00020$ at 50° ($v=9$). Neither hydrochloric acid nor acetic acid influences the rate of decomposition of solutions of pure *p*-nitrodiazobenzene chloride to any marked degree (compare Schwalbe, *loc. cit.*).

Diazo-acetates in aqueous solution do not decompose in a normal manner; the weaker the diazo-base the more abnormal is the course of the reaction. As was stated by Schwalbe, the presence of sodium chloride increases the stability of solutions of *p*-nitrodiazobenzene acetate.

Solutions of diazo-salts are slowly decomposed by nitrous acid (compare Cain, *loc. cit.*); the nitrous acid does not act as a catalyst, as stated by Schwalbe, but slowly disappears, since it is directly concerned in the decomposition.

W. H. G.

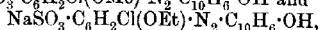
[Preparation of Disazo-derivatives of 2-Amino-*p*-cresol.]
ANILINFARBEN & EXTRACT FABRIKEN VORM. J. R. GEIGY (D.R.P. 201377).—2-Amino-*p*-tolyl toluene-*p*-sulphonate, $C_7H_7SO_3 \cdot O \cdot C_7H_7NH_2$,

lustrous prisms, m. p. 78°, is obtained by reducing 2-nitro-*p*-tolyl toluene-*p*-sulphonate; it couples with diazonium salts and diazoaryl-sulphonic acids, yielding diazotisable *p*-aminoazo-derivatives, such as sodium sulphobenzene-5-azo-2-amino-*p*-tolyl toluene-*p*-sulphonate,
 $\text{SO}_3\text{Na}\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{C}_6\text{H}_5(\text{NH}_2)\cdot\text{O}\cdot\text{SO}_2\cdot\text{C}_7\text{H}_7$.

The diazo-compounds of these *p*-aminoazo-derivatives couple with naphthol in alkaline solution, and the resulting disazo-derivatives give valuable colouring matters on removing the toluene-*p*-sulphonic group by hydrolysis.

G. T. M.

Azo-derivatives of 2-Chloro-*p*-anisidine- and 2-Chloro-*p*-phenetidine-sulphonic Acids. ACTIEN-GESELLSCHAFT FÜR ANILIN-FABRIKEN (D.R.-P. 189469).—Insoluble coloured salts of the azo-compounds, $\text{NaSO}_3\cdot\text{C}_6\text{H}_4\text{Cl}(\text{OMe})\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_7\cdot\text{OH}$ and



are produced by double decomposition with barium or other metallic chlorides. When mixed with aluminium hydroxide, these substances give rise to scarlet lakes of great brilliancy and fastness. G. T. M.

Azo-derivatives of Phenol and of the Phenolcarboxylic Acids. EUGENE GRANDMOUGIN and HANS FREIMANN (*J. pr. Chem.*, 1908, [ii], 78, 384—408).—A continuation and extension of former work (compare Abstr., 1907, i, 664, 986, 987; this vol., i, 926).

Azo-derivatives of Phenols.—The benzenesulphonyl ester of *p*-hydroxyazobenzene crystallises in yellow needles, m. p. 109°. The acetyl derivative of bis-benzeneazophenol has m. p. 116°, and the benzenesulphonyl ester forms yellowish-brown needles, m. p. 99°. The benzenesulphonyl ester of tris-benzeneazophenol forms red crystals, m. p. 202°.

o-Tolueneazophenol has m. p. 107—108° (Noelting and Werner, Abstr., 1891, 212, give 102—103°; Paganini, Abstr., 1891, 556, gives 101°); the acetyl derivative crystallises in reddish-yellow leaflets, m. p. 68°, and the benzenesulphonyl ester forms small, orange leaflets, m. p. 64°. Bis-*o*-tolueneazophenol has m. p. 116° (Noelting and Werner, *loc. cit.*, give 146°; Paganini, *loc. cit.*, gives 116°). The acetyl derivative forms reddish-yellow needles, m. p. 75—76°. When bis-*o*-tolueneazophenol is prepared from diazotised *o*-toluidine and ethyl *p*-hydroxybenzoate, it has m. p. 150°, and the acetyl derivative has m. p. 98°. The nature of this difference is not yet known.

Bis-*m*-tolueneazophenol forms yellowish-brown needles, m. p. 94°; the acetyl derivative crystallises in yellow needles, m. p. 75°. Tris-*m*-tolueneazophenol, prepared by the action of *m*-diazotoluene on salicylic acid, forms brown needles, m. p. 160°. The acetyl derivative of *p*-tolueneazophenol has m. p. 95°; the benzenesulphonyl ester forms brownish-yellow leaflets, m. p. 114°. Bis-*p*-tolueneazophenol has m. p. 176°, and the acetyl derivative has m. p. 138° (Goldschmidt and Pollak, Abstr., 1892, 974, give 170° and 128° respectively). Tris-*p*-tolueneazophenol forms brownish-red needles, m. p. 238°; the acetyl derivative crystallises in golden needles, m. p. 187°. 2-*p*-Nitrobenzene-azo-4-benzeneazophenol, prepared by treating *p*-hydroxyazobenzene with

diazotised *p*-nitroaniline, has m. p. 196°; the *acetyl* derivative has m. p. 189°. 4-*p*-Nitrobenzeneazo-2-benzeneazophenol, prepared from *o*-hydroxyazobenzene and diazotised *p*-nitroaniline, has m. p. 189°; the *acetyl* derivative has m. p. 154°. 4-Acetylamino-2-*p*-nitrobenzeneazophenol, prepared by the interaction of *p*-nitrodiazobenzene and *p*-acetylamino-phenol, forms brown needles, m. p. 227°; it is hydrolysed by 60% sulphuric acid to 4-amino-2-*p*-nitrobenzeneazophenol, golden crystals, m. p. 211°. β -Naphthaleneazophenol forms bronze needles, m. p. 240°; the *acetyl* derivative crystallises in brown needles, m. p. 180°.

Azo-derivatives of Salicylic Acid.—Methyl *p*-nitrobenzeneazosalicylate forms silky, orange needles, m. p. 166°; the *acetyl* derivative forms orange-yellow needles, m. p. 131°. The *acetyl* derivative of phenyl benzeneazosalicylate (Limpricht, Abstr., 1891, 1036) forms golden needles, m. p. 132°. Limpricht's phenyl bis-benzeneazosalicylate is hydroxyazobenzene. Phenyl *p*-nitrobenzeneazosalicylate crystallises in yellow needles, m. p. 165°; the *acetyl* derivative forms yellow needles, m. p. 155°. *o*-Nitrobenzeneazosalicylaldehyde forms brown crystals, m. p. 141°; the phenylhydrazone forms brown needles, m. p. 192°. Methyl bis-benzeneazosalicylate forms brown crystals, m. p. 141°; the *acetyl* derivative forms yellowish-red needles, m. p. 145°. Methyl bis-*o*-tolueneazosalicylate forms brownish-yellow needles, m. p. 154°. Bis-*m*-tolueneazosalicylic acid forms brownish-olive needles, m. p. 210°; the *acetyl* derivative forms yellow needles, m. p. 170°. Bis-*p*-tolueneazosalicylic acid crystallises in brown, felted needles, m. p. 214°; the *acetyl* derivative forms reddish-brown tablets, m. p. 195°. These bisazo-derivatives of salicylic acid are all prepared in alkali hydroxide solution.

Azo-derivatives of p-Hydroxybenzoic Acid.—The *acetyl* derivative of benzeneazo-*p*-hydroxybenzoic acid has m. p. 205°, not 198–199° as previously given. *o*-Tolueneazo-*p*-hydroxybenzoic acid crystallises in orange-yellow needles, m. p. 223°. In the preparation of this substance from diazotoluene chloride and *p*-hydroxybenzoic acid in alkali hydroxide solution, there are also formed bis-*o*-tolueneazophenol and *o*-tolueneazoinazole; the *acetyl* derivative of the latter forms golden-yellow needles, m. p. 163°, and the benzoyl derivative, yellow needles, m. p. 179.5°. *p*-Tolueneazo-*p*-hydroxybenzoic acid forms yellow needles, m. p. 236°; the *acetyl* derivative forms yellow needles, m. p. 205°.

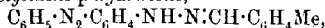
Ethyl benzeneazo-*p*-hydroxybenzoate (Anwers and Röhrig, Abstr. 1897, i, 341) is readily prepared by the action of diazobenzene chloride on ethyl *p*-hydroxybenzoate; the *acetyl* derivative forms yellowish orange needles, m. p. 137°. Ethyl *o*-tolueneazo-*p*-hydroxybenzoate has m. p. 105°.

Azo-derivatives of m-Hydroxybenzoic Acid.—Benzeneazo-*m*-hydroxybenzoic acid has m. p. 221° (Limpricht, loc. cit., gives 213°); the methyl ester forms brown needles, m. p. 78–79°. Bis-benzeneazo-*m*-hydroxybenzoic acid occurs in two isomeric forms, namely, the 2:6 and the 4:6-bis-benzeneazo-derivatives. Of these, the α -acid (not specified) forms brown needles, m. p. 222–223°, and the β -acid has m. p. 226–227°; it is sparingly soluble in methyl alcohol, whilst the

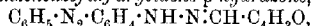
acid is readily soluble in this solvent. The *methyl ester* of the β -acid forms bronze-yellow, felted needles, m. p. 183°. J. C. C.

Azobenzene-*p*-hydrazinesulphonic Acid resulting from the Action of Sulphur Dioxide on Diazobenzene Sulphate and its Condensation with Aldehydes and Ketones. JULIUS TRÖGER and O. MÜLLER (*J. pr. Chem.*, 1908, [ii], 78, 369—383).—In continuation of previous work (Tröger, Berlin, and Franke, *Abstr.*, 1906, i, 994), the authors have prepared a further number of hydrazones by condensing azobenzene-*p*-hydrazinesulphonic acid with aldehydes and ketones in the presence of glacial acetic acid or alcoholic hydrochloric acid.

Benzeneazo-p-tolylidene-p-hydrazone,

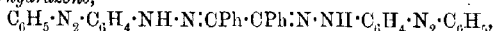


prepared from *p*-tolualdehyde, crystallises in orange-yellow needles, m. p. 178°; the *hydrochloride* forms small, blue needles. *Benzeneazo-cumylidene-p-hydrazone*, $\text{C}_6\text{H}_5\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{N}:\text{CH}\cdot\text{C}_6\text{H}_4\cdot\text{C}_3\text{H}_7$, prepared from cuminaldehyde, forms yellowish-red needles, m. p. 144—145°; the *hydrochloride* is dark blue. *Benzeneazo-m-chlorobenzylidene-p-hydrazone*, $\text{C}_6\text{H}_5\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{N}:\text{CH}\cdot\text{C}_6\text{H}_4\text{Cl}$, prepared from *m*-chlorobenzaldehyde, forms small, glistening, golden-yellow leaflets, m. p. 160.5°; the *sulphate* forms dark blue needles. *Benzeneazo-m-bromobenzylidene-p-hydrazone* crystallises in small, glistening, golden-yellow leaflets, m. p. 173°; the *sulphate* is blue. *Benzeneazo-p-aminobenzylidene-p-hydrazone*, prepared from *p*-aminobenzaldehyde, forms small, reddish-brown leaflets, darkening at 136°, and decomposing above this, the residue being completely melted at 180.5°. *Benzeneazo-p-dimethylaminobenzylidene-p-hydrazone* crystallises in cinnamon-red needles, m. p. 185.5°; the *hydrochloride* forms small, glistening, blue needles. *Benzeneazo-p-hydroxybenzylidene-p-hydrazone* forms dark red needles, m. p. 196°; the *hydrochloride* consists of dark blue needles. *Benzeneazofurfurylidene-p-hydrazone*,



prepared from furfuraldehyde, forms reddish-brown tablets, m. p. 133°; the *hydrochloride* is brownish-black.

Benzeneazobenzophenone-p-hydrazone, $\text{C}_6\text{H}_5\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{N}:\text{CPh}_2$, prepared from benzophenone, forms brownish-red leaflets, m. p. 144°; the *hydrochloride* forms blue needles or plates. *Benzeneazobenzil-p-hydrazone*,

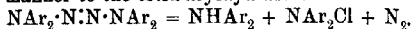


prepared from benzil, forms orange-red needles, m. p. 184—185°; the *hydrochloride* is blue. *Benzeneazobenzylidene-p-hydrazone hydrochloride* is a black powder. J. C. C.

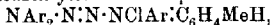
Trisbenzeneazophenol. LÉO VIGNON (*Bull. Soc. chim.*, 1908, [iv], 3, 1030—1031).—In previous work (*Abstr.*, 1904, i, 699) on bisbenzeneazophenol, the author obtained indications of the formation of a substance richer in nitrogen, and this has led him to repeat Grandmougin and Freimann's work on trisbenzeneazophenol (*Abstr.*, 1907, i, 664). He confirms their results, but points out that the yield of the trisazo-substance is only 1% of the theoretical.

T. A. H.

Aromatic Tetrazens. V. HEINRICH WIELAND (*Ber.*, 1908, 41, 3498—3506).—The tetrazens are decomposed by hydrogen chloride in a similar manner to the tetra-arylhydrazines:



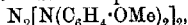
Thus tetratolyltetrazen yields first a quinonoid salt,



indicated by a violet coloration in solution, which is decomposed with evolution of nitrogen, and finally *p*-ditolylamine, tolazonium and dichlorotolazonium chloride result. Tetra-anisyltetrazen behaves similarly; acetic acid brings about this decomposition in the cold with remarkable ease.

p-Tetratolyltetrazen, $\text{N}(\text{C}_7\text{H}_7)_2 \cdot \text{N} : \text{N} \cdot \text{N}(\text{C}_7\text{H}_7)_2$, prepared by oxidation of *p*-ditolylhydrazine, preferably with permanganate, crystallises in glistening, yellow needles, decomp. 134° . It is decomposed by hydrogen chloride or acetic acid to tolazine derivatives. With bromine, a bluish-violet coloration indicates a primary additive product, which changes to a brownish-green; but little ditolylamine along with much dibromotolazine is formed.

To prepare *p*-dianisylhydrazine, *p*-dianisylamine is converted into the *nitrosoamine*, crystallising in flat, bright yellow needles, m. p. 79° , and dissolving in concentrated sulphuric acid with a greenish-blue coloration. On reduction with zinc dust and acetic acid, the *dianisylhydrazine*, $\text{NH}_2 \cdot \text{N}(\text{C}_6\text{H}_4 \cdot \text{OMe})_2$, is obtained in mother-of-pearl-like, broad pointed, colourless needles, m. p. 111° . The *hydrochloride* is sparingly soluble. The oxidation to *p*-tetra-anisyltetrazen,



could only be effected by means of *p*-benzoquinone. The tetrazen forms citron-yellow, four-sided plates, m. p. 117° (violent decomp.). Decomposition with acids gives the characteristic anisazonium compounds.

Tetratolyltetrazen dissolved in xylene, when heated for a few minutes, shows a violent evolution of nitrogen and formation of tetratolyldiazine. E. F. A.

Catabolism of Proteins. FRANZ HOFMEISTER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 273—281).—This paper is mainly a review of recent work on the cleavage of proteins, and the importance of such work and the necessity of dealing with pure substances are among the points insisted upon. W. D. H.

Effect of Acid and Alkali on the Osmotic Pressure of the Serum Proteins. L. ADAMSON and HERBERT E. ROAF (*Bio-Chem. J.*, 1908, 3, 422—438).—Alkali increases (although not proportionally to the amount added) the osmotic pressure of the serum proteins. Acid causes this pressure to fall to zero, but larger amounts of acid increase it again. The point of zero pressure corresponds with the "alkaline reactivity" of serum ash. When the amount of alkali added is corrected by this "alkaline reactivity," the relationship of the osmotic pressure to the amount of alkali is a logarithmic one. The experiments with alkali are limited by the breaking up of the protein into particles small enough to pass a parchment paper membrane, and with acid by the partial precipitation of the protein. W. D. H.

Dissociation of Solutions of the Basic Caseinates [Caseinogenates] of Sodium and Ammonium. T. BRAILSFORD ROBERTSON (*J. Physical Chem.*, 1908, 12, 473—483).—In a previous paper (*Abstr.*, 1907, i, 1096), it has been shown that the solutions of sodium and ammonium caseinate neutral to litmus obey Ostwald's dilution law for binary electrolytes. Sackur (*Abstr.*, 1903, ii, 4) had previously carried out electrical conductivity measurements with solutions of the corresponding "basic" salts, which were obtained by adding sufficient alkali to make the solutions neutral to phenolphthalein, the amounts of alkali required to produce the basic and neutral solutions being in the ratio 8:5. The author now shows from Sackur's results that the solutions of the basic caseinates also follow the dilution law for binary electrolytes, so that casein appears to behave as a monobasic acid in both solutions. Sackur has suggested that casein behaves to alkalis as a tetrabasic acid, but it is shown that the evidence for this view is untrustworthy.

The above results are best accounted for on the view that complex ions containing casein are present. The neutral caseinates are amphoteric salts of the combined base, containing the ions NaX' and XXOH' ($\text{X} = \text{casein}$), whilst the basic caseinates are true salts of the combined base, the solutions containing the ions Na' and XXOH' . In all the solutions there is an equilibrium of the type $\text{NaX}' + \text{HXOH} \rightleftharpoons \text{Na}' + \text{HXXOH}$, which is not displaced by dilution.

From Sackur's results, the value 15.2×10^{-5} cm./sec. is obtained for the velocity of the casein ion in solutions of basic sodium caseinate, and 9.1×10^{-5} cm./sec. for the velocity of the same ion in solutions of ammonium caseinate.

The dissociation constants for the "basic" sodium and ammonium salts of casein are 0.0499 and 0.0404 respectively. G. S.

Action of Iron and of Heavy Metals [as Oxygen Carriers]. VINCENZO CERVELLO (*Arch. exp. Path. Pharm., Suppl.*, 1908, 107—109).—The oxidation of guaiacum resin is accelerated by the salts of heavy metals, and most of all by those of iron and copper. In the case of ferric salts, a partial reduction to the ferrous state occurs. The part played by iron as a constituent of hæmoglobin is discussed. G. B.

Urochrome. I. HERMANN HOHLWEG (*Biochem. Zeitsch.*, 1908, 13, 199—204).—The pigment was prepared from human urine, which was made alkaline with calcium hydroxide, precipitated with calcium chloride, the filtrate neutralised with acid, and evaporated in a vacuum to a syrup; after separation of the greater part of the salts, the syrup was diluted and shaken for several hours with animal charcoal, which absorbed the pigment, and from which it was afterwards removed by glacial acetic acid. From the solution thus obtained, it was precipitated by ether. The brown powder prepared in this way contained 8.22% of ash, and was soluble in water and methyl alcohol, but insoluble in strong ethyl alcohol and other organic solvents. Its spectrum showed no absorption bands, and it did not give the urobilin reaction with ammoniacal zinc chloride. It gave the

Garrod reaction with acetaldehyde and subsequent treatment with ammoniacal zinc chloride; also the Molisch reaction with α -naphthol.
S. B. S.

Urochrome. II. K. E. SALOMONSEN (*Biochem. Zeitsch.*, 1908, 13, 205—207).—The product was prepared by a slight modification of Hohlweg's method (preceding abstract). It was purified by conversion into its calcium salt. On treatment of an aqueous solution of the pigment with bromine in the cold, a yellow bromo-derivative was obtained, which was soluble in alkalis. On warming an aqueous solution of urochrome with bromine, a more vigorous bromination accompanied by oxidation takes place.
S. B. S.

Urochrome. III. STEFANO MANCINI (*Biochem. Zeitsch.*, 1908, 13, 208—214).—The bromo-derivative of Hohlweg and Salomonsen (preceding abstracts) was prepared by a modified method, unconcentrated urine being employed for the purpose. It was purified by dissolving in alkali, precipitating by acid, dissolving the substance thus obtained in water, and treating again with bromine. The doubly-refracting, yellow product thus prepared, when heated with zinc dust or calcium hydroxide, gives strong pyrrole reactions. Its composition corresponds approximately with the formula $C_{30}H_{41}O_{12}N_7Br_6$, which would be a bromo-derivative of $C_{38}H_{47}O_{12}N_7$. This is not far removed from the formula $C_{36}H_{39}O_{16}N_7$, which Thudichum ascribed to uromelanin. For it the name *uropyrryl* is proposed. By oxidation of crude bromouropyrryl with bromine water, oxalic acid, an oily acid, which gives the pyrrole reaction, and also, most probably, bromoanil were obtained. Neither oxalic acid nor bromoanil were obtained when a product, purified by repeated solution in alkali, re-precipitation with acids, and re-bromination, was employed. The relation of *uropyrryl* to urochrome is not yet ascertained.
S. B. S.

Hippomelanin. I. PETER RONA and O. RIESSER (*Zeitsch. physiol. Chem.*, 1908, 57, 143—153).—Hippomelanin, purified by Fürth and Jerusalem's method (Abstr., 1907, ii, 797), has been oxidised by a 3% solution of hydrogen peroxide containing a small amount (0.002%) of hydrochloric acid in solution. The total nitrogen of the aqueous solution was 7.5%, and the ammoniacal nitrogen 4.2%, that is, 56% of the total nitrogen present in the hippomelanin. Attempts were made to isolate the other nitrogenous products in the form of copper compounds. A copper derivative was obtained which was insoluble in alcohol and water. This contained a small amount of copper oxalate, but no other constituent was obtained in a pure form.

A copper derivative soluble in alcohol and water was also obtained. From this a base, which was precipitated by phosphotungstic acid, was isolated. The *hydrochloride* is readily soluble in water or alcohol. The *picrate* is deposited as an oil sparingly soluble in alcohol; the *picrolonate* is a deep red compound sparingly soluble in water, and the *platinichloride*, $N = 16.14\%$, forms ochre-yellow prisms, m. p. 280° , insoluble in alcohol.
J. J. S.

Inosic Acid. CARL NEUBERG and B. BRAHN (*Ber.*, 1908, 41, 3376—3381. Compare Abstr., 1907, i, 1097).—It is pointed out that the sugar obtained by hydrolysing inosic acid cannot be *r*-arabinose, as stated by Bauer (Abstr., 1907, i, 1098). The authors confirm their view that it is *l*-xylose. They have obtained it in a syrupy form, have shown that it gives the pentose reactions, and that it is dextrorotatory. They further show that it does not yield a diphenylhydrazone as readily as does *r*-arabinose.

Levene and Jacob's conclusion (this vol., i, 931), that in place of a pentose inosic acid yields a tetrose-carboxylic acid, is shown to be untenable. J. J. S.

Glucothionic Acid. JOHN A. MANDEL and CARL NEUBERG (*Biochem. Zeitsch.*, 1908, 13, 142—147. Compare Abstr., 1905, ii, 736).—Preliminary investigations show that the product obtained from the kidneys contains sulphuric acid in a conjugated form. Dextrose is not a product of hydrolysis, and neither is the substance of polysulphide nature. For these reasons, the name glucothionic acid is inapplicable. Names indicating the origin of the product are suggested, for example, *renosulphuric acid*. S. B. S.

Jecorin and other Lecithin-like Substances in the Liver of the Horse. A. BASKOFF (*Zeitsch. physiol. Chem.*, 1908, 57, 395—460).

Jecorin was prepared so that it gave a constant N:P ratio and a constant yield of sugar (about 14%). It is a diamino-monophosphatide in Erlandsen's sense.

The alcoholic extract of the liver is evaporated to a syrupy consistency, and treated with ether; a jecorin-like substance forms as a heavy layer at the bottom of the vessel; there is also a brown precipitate, insoluble in ether and alcohol, which was not further investigated. The addition of alcohol to the ether extract produces a precipitate of crude jecorin, lecithin remaining in solution. The precipitate is again treated with ether, and a jecorin-like substance remains insoluble; the extract, however, contains Drechsel's jecorin, which is precipitable by alcohol, and can be purified by the repetition of this process three or four times. W. D. H.

Glutamic Acid in Various Keratins. EMIL AUERHAIDEN and BENES FUCHS (*Zeitsch. physiol. Chem.*, 1908, 57, 339—341).—The following table gives the mean analytical results obtained, in parts %:

	Hoof of ox.		Horn of ox.		Hoof of horse.
	1 yr. old.	4 yrs. old.	1 yr. old.	4 yrs. old.	
Dry residue	90.5	91.5	96.0	96.5	75.4
N	0.14	0.16	0.22	0.36	0.45
Melanin substances	0.22	0.12	1.65	0.96	0.9
Glutamic acid hydrochloride in ash-free dry residue.....	18.0	16.8	13.8	12.9	18.2

Attention is called to the variable numbers obtained, and to the fall of glutamic acid and rise of ash with increasing age. W. D. H.

The Composition of the Protamine from Salmon Spermatozoa. LOUIS NELSON (*Arch. exp. Path. Pharm.*, 1908, 59, 331—335).—The protamine was prepared by Schmiedeberg's method, and converted into the platinichloride. It was found that an impurity could be removed from the latter by hot water. The protamine was found then to have a composition corresponding with the formula $C_{16}H_{27}O_5N_7$.
S. B. S.

Chemistry of Protamines. I. Protamine from the Spermatozoa of the Caspian Sturgeon, *Accipenser Guldenstadti*. W. D. MALENÜCK (*Zeitsch. physiol. Chem.*, 1908, 57, 99—112. Compare Kuračeff, Abstr., 1901, ii, 462).—For isolating protamines and determining the yield, the method of precipitating with sodium picrate is preferred to Kossel's method of repeated solution of the sulphate in water and precipitation with alcohol. The sulphuric acid extract is stirred continuously with the aqueous picrate solution until a flocculent, orange-yellow precipitate is formed. The filtrate should be clear and free from opalescence, and should yield a further precipitate with sodium picrate. The precipitated picrate is washed, dried, dissolved in acetone, and decomposed with sulphuric acid. A portion of the picrate is insoluble in acetone, and this is shown to consist of adenine picrate, whereas the protamine picrate is readily soluble. In decomposing the acetone solution of the picrate, it is essential that an excess of sulphuric acid is not added.

The last traces of picric acid can be removed by solution in water, evaporating the solution, carefully precipitating with alcohol and acetone, and rubbing the sticky mass in a mortar with acetone until hard. The dry sulphate prepared by such a method usually contains only 0.3% of ash when dried at 108—110°, and gave the following analytical results: C, 37.64; H, 6.61; N, 20.92, and H_2SO_4 22.59%. Similar data were obtained with a sulphate which had been converted into the chloride by Goto's method. The formula $C_{27}H_{55}O_7N_{13} \cdot 2H_2SO_4$ agrees best with these data.
J. J. S.

Thymamine, a Protamine from the Thymus Gland. LOUIS NELSON (*Arch. exp. Path. Pharm.*, 1908, 59, 336—340).—The protamine was prepared by extracting the coagulated proteins of the gland with copper chloride solution by Schmiedeberg's method, by means of which a solution of the hydrochloride is obtained. Sodium hydroxide is added until the reaction is neutral, and the filtrate from the precipitate thus produced evaporated, when copper protein combinations separate. The filtrate from these is diluted and evaporated, and more protein separates and is filtered off. These processes are repeated until a solution which no longer gives a reaction with Millon's reagent is obtained. The protamine is prepared from this by precipitating as phosphotungstate, and is finally obtained in the form of a platinichloride, the purification of which is described in detail. Its composition corresponds with the formula $C_{22}H_{40}O_5N_6$.
S. B. S.

The Action of Tyrosinase from *Russula delica* on Polypeptides which contain Tyrosine and on Suprarenine. EMIL ABDEKHALDEN and MARKUS GUGGENHEIM (*Zeitsch. physiol. Chem.*, 1908, 57,

329—331. Compare this vol., i, 237).—In addition to the peptides previously used, tyrosinase rapidly splits *d*-alanyl-*L*-tyrosine and *L*-leucyl-*L*-tyrosine. It also decomposes suprarenine (adrenaline), with the rapid formation of a red colour, and later of dark-coloured flocculi; *L*-, *d*-, and *DL*-suprarenines are all decomposed with equal rapidity.

W. D. H.

Milk Proteins and Enzymes. GEORGE A. OLSON (*J. Biol. Chem.*, 1908, 5, 261—282).—After removal of the caseinogen and albumin, milk, cream, and butter are stated to yield a new protein, which is precipitable by hydrochloric acid. It contains 18.9% of nitrogen. When it is added to milk, it produces partial digestion of the caseinogen; this power is most active at 65°, and is destroyed at 80°. After removal of the new protein, the filtrate also has the same property; the ferment action of the protein is therefore believed to be due to incorporation.

W. D. H.

The Scission by Diastase of Lactose, Maltose, and their Derivatives. H. BIERRY and J. GIAJA (*Compt. rend.*, 1908, 147, 268—270).—The lactone of lactobionic acid was treated with macerations of foetal intestines (sheep and oxen) and with digestive juices of snails. The former was inactive, whereas the latter caused the production of galactose; it also acts on lactosazone with the production of galactose. Similar results were obtained with the lactone of maltobionic acid. These facts afford further evidence of the specific nature of the ferments.

S. B. S.

Action of Organo-magnesium Compounds on Arsenious Oxide. FRANZ SACHS and HANS KANTOROWICZ (*Ber.*, 1908, 41, 1767—1769).—The addition of powdered arsenious oxide to an ethereal solution of magnesium phenyl bromide leads to the formation of diphenylarsine oxide after thirty minutes' heating, and of triphenylarsine after three hours. With magnesium *p*-tolyl bromide, only tri-*p*-tolylarsine is obtained, whilst with magnesium benzyl bromide, after three hours' heating, a substance, $\text{As}(\text{CH}_2\text{Ph})_2 \cdot \text{OH} \cdot \text{H}_2\text{O}$, m. p. 15—216°, is formed after the usual operations.

C. S.

Triphenylstibine Sulphide. LUDWIG KAUFMANN (*Ber.*, 1908, 41, 762—2766).—Contrary to the statement of Michaelis and Rees (*Abstr.*, 1886, 884), triphenylstibine sulphide can be prepared under the following conditions: Pfeiffer's triphenylstibine (*Abstr.*, 1905, i, 64) is converted into a haloid additive compound, preferably the iodide, which is dissolved in alcoholic ammonia, and hydrogen sulphide passed through the solution until a faint yellow coloration appears in the liquid; the crystals which have separated are recrystallised from alcohol, whereby colourless triphenylstibine sulphide, SbPh_3S , is obtained, m. p. 119—120°. Prolonged heating of its solutions or treatment with acids, even acetic, causes decomposition into sulphur and triphenylstibine.

If, during the preparation, the current of hydrogen sulphide is continued, the crystals redissolve to a yellowish-red solution, which

probably contains a very labile antimony compound, for, by keeping, the liquid deposits sulphur, and the colourless filtrate, which is free from sulphur, yields pure triphenylstibine by evaporation. C. S.

Action of Grignard Reagents on Ethyl Orthosilicate.

EUGEN KHOTINSKY and BASILE SEREGENKOFF (*Ber.*, 1908, 41, 2946—2953).—When ethyl orthosilicate, dissolved in dry ether, is added gradually to a cold ethereal solution of a Grignard reagent, the mixture heated for ten to fifteen minutes on the water-bath, and decomposed by 50% acetic acid, compounds are obtained of the type $R \cdot Si(OEt)_2$. Only one ethoxyl group can be replaced (compare Tschitschibabin, *Abstr.*, 1905, i, 283). If the heating is prolonged, or if excess of the Grignard reagent is used, complex silicon compounds are formed. In order to prevent the presence of any trace of moisture, the reaction is carried out in a current of dry hydrogen.

Ethyl orthosilicobenzoate, $SiPh(OEt)_3$, b. p. 232—234°, has been prepared by Ladenburg (*Annalen*, 1874, 173, 151); it is hydrolysed by cold hydriodic acid, D 1.7, yielding Ladenburg's silicobenzoic acid, $C_6H_5 \cdot SiO_2H$, m. p. 92°, which is also obtained when the reaction product of an excess of magnesium phenyl bromide and ethyl orthosilicate (1 mol.) is decomposed by sulphuric acid. By prolonged heating of the mixture before decomposition by sulphuric acid, trisilicobenzoysilicic acid, $OH \cdot Si(O \cdot SiPhO)_3$, is obtained, which is also found in the high-boiling residue in the preparation of the ortho-ester; it appears to be identical with Polis' compound (*Abstr.*, 1886, 618). Ethyl ortho-2:4-dimethylsilicobenzoate, $C_6H_3Me_2 \cdot Si(OEt)_3$, b. p. 268—271°, obtained in a similar manner from magnesium *m*-xylyl iodide, yields, by hydrolysis, the acid, $C_6H_3Me_2 \cdot SiO_2H$, m. p. 118—121°. The residue in the distillation of the ester contains trisilico-xylylsilicic acid, $Si(O \cdot SiO \cdot C_6H_3Me_2)_3 \cdot OH$, an infusible, non-volatile, greyish-white powder.

Ethyl α -orthosiliconaphthoate, $C_{10}H_7 \cdot Si(OEt)_3$, has b. p. 293—295°, and yields, by hydrolysis with cold hydriodic acid, α -siliconaphthoic acid, $C_{10}H_7 \cdot SiO_2H$, m. p. 239°, softening at 225°, and, by hydrolysis with cold concentrated sulphuric acid, a sulpho- α -siliconaphthoic acid, $SO_3H \cdot C_{10}H_7 \cdot SiO_2H$, m. p. 88—90°. Ethyl β -orthosiliconaphthoate, $C_{10}H_7 \cdot Si(OEt)_3$, has b. p. 270—273°, and the acid, $C_{10}H_7 \cdot SiO_2H$, m. p. 248—250°. C. S.

A Zirconium Mercury Double Salt. WALTER PETERS (*Ber.*, 1908, 41, 3173—3175).—Although zirconium tetrachloride and mercury phenyl may be heated in a closed tube or under a pressure of 1 mm. without undergoing change, interaction takes place if the tube is evacuated by a water pump, owing to the presence of water vapour. The white, hygroscopic needles of the salt, $ZrOCl_2 \cdot 2HgClPh$, are soluble in ether, and, when submitted to vacuum distillation at 170—210°, give a sublimate of mercury phenyl chloride and a residue of the composition $Zr_3O_4Cl_4 \cdot 6HgClPh$. Mercury ethyl and mercury *o*-tolyl are without action. W. R.

General and Physical Chemistry.

Ultraviolet Spectrum of Silicon. ANTOINE DE GRAMONT and CHARLES DE WATTEVILLE (*Compt. rend.*, 1908, 147, 239—242).—The ultraviolet spectrum of silicon containing both lines and bands has been mapped between $\lambda 2123.0$ and $\lambda 2987.8$. The spectrum of the oscillating spark passing between silicon poles in hydrogen in a quartz tube gave 23 lines and 14 bands, whereas the flame spectrum obtained by burning ordinary gas impregnated with silicon chloride vapour contained 26 bands and 11 lines. The relative intensities of lines and bands common to both spectra were not the same.

The oscillation frequencies of the bands seem to fall into series, but the heads are ill-defined. R. J. C.

Dispersion in the Electric Spectra of Benzene, Toluene, and Acetone. A. R. COLLEY (*J. Russ. Phys. Chem. Soc.*, 1908, 40 [*Phys.*], 228—244).—The author has investigated the electrical spectra of benzene, toluene, and acetone, a method being used which permits of accurate measurements with small troughs in the case of liquids with low refractive indices. The results, which are given in the form of tables and curves, bring out the intimate chemical relationship between toluene and benzene, two bands being common to the two spectra. Further, the spectra of toluene and acetone also exhibit one common band, which must be regarded as typical for the methyl group. T. H. P.

Discontinuous Cathode Luminescence Spectra of Some Aromatic Compounds. OTTO FISCHER (*Zeitsch. wiss. Photograph. Photochem.*, 1908, 6, 305—326. Compare Goldstein, *Abstr.*, 1904, ii, 689, 690).—The emission spectra of the substances exposed to cathode rays at the temperature of solid carbon dioxide were photographed. Difficulties were found in certain cases, owing to the rapid change of colour of some substances under the influence of cathode rays. The ultraviolet spectra were not examined.

The number of luminous bands is with most aromatic substances considerable. In all the cases examined, the sharp edges of the bands are turned towards the red, the blue side being more or less indistinct. It is frequently possible to group the bands in series, corresponding pairs of which show a constant difference of wave-length.

In the case of naphthalene compounds, blue and violet light is only emitted for a short time, the colour changing to yellow or red. Three characteristic blue bands of naphthalene reappear in the spectra of all the naphthalene derivatives examined, and also in those of diphenyl and phenanthrene, but, whilst the last two compounds continue to emit these bands, the structure producing them is rapidly destroyed by the bombardment in the case of naphthalene derivatives. The same

bands appear, strongly shifted towards the red, in anthracene and dibromoanthracene.

C. H. D.

Influence of Temperature and of the State of Aggregation on the Absorption Spectra of Molten Salts. T. RETSCHINSKY (*Ann. Physik*, 1908, [iv], 27, 100—112).—An examination of the absorption spectra of some thirty molten inorganic salts has shown that, in all cases, there is a well-defined limit to the absorption band on the ultra-violet side of the spectrum, all radiations of smaller wave-length than that corresponding with the limiting value being completely absorbed. With rise of temperature, this limit is displaced in the direction of greater wave-lengths. When the displacement is expressed in terms of wave-lengths, its magnitude, for a given rise of temperature, increases with the wave-length; when expressed in terms of frequencies, the displacement is independent of the wave-length. For a temperature rise of 100° , the absorption limit is displaced to an extent represented by 10 to $20 \times 10^3/\mu\mu$.

Some of the salts have been also investigated in the solid state. The influence of temperature on the absorption limit is of the same order of magnitude as in the case of the molten substances. On liquefaction, the absorption limit is displaced considerably in the case of the chlorides and bromides of potassium and sodium, but no sharp change is met with in the case of silver nitrate, silver chloride, or lead chloride.

In regard to the influence of chemical composition on the absorption, it is found that the salts of the heavy metals absorb more than those of the alkali and alkaline-earth metals. In general, for a given metal the sulphate absorbs most light; then follow the chloride, bromide, iodide, and nitrate with gradually decreasing absorptions. For salts of the same acid containing metals belonging to the same group of the periodic system, the absorption limit is nearer the red end of the spectrum the greater the atomic weight of the metal.

H. M. D.

Fluorescence of Sodium Vapour. HANS ZICKENDRANT (*Physikal. Zeitsch.*, 1908, 9, 593—603).—The thermal and electrical condition of fluorescing sodium vapour has been examined. In contact with air, fluorescence begins at 280 — 290° ; this temperature is independent of the pressure, although the sharpness with which the phenomenon sets in is much greater at higher pressures than at low. Measurements have been made of the minimum potential required for electrical discharge through the vapour under different conditions. For sodium vapour in contact with air, the potential temperature curves show minima at 240° and 280° ; the latter is supposed to correspond with the commencement of fluorescence. In contact with nitrogen and carbon dioxide, the minimum potential differences corresponding with this are 300° and 340° respectively. In contact with hydrogen, an olive-coloured fluorescence can be observed at 370° , but no current could be passed through the vapour with the available potential of 400 volts.

The properties of the vapour in contact with air have been examined in greater detail. Curves are plotted which show the dependence of the current on the discharge potential under different pressures, and also the dependence of the potential on the pressure and on the temperature for a given current intensity. When a current has been passed through the vapour for some time, a smaller potential is required for discharge immediately after the cessation of the current than when some time has elapsed. The potential gradually increases, and attains a constant value after some minutes. The paper concludes with a theoretical discussion of the cause of the conducting power of sodium vapour.

H. M. D.

Fluorescence of Organic Substances. JOHANNES STARK and W. STEUBING (*Physikal. Zeitsch.*, 1908, 9, 661—669. Compare Abstr., 1907, ii, 147, 417; this vol., ii, 138, 746).—Further observations relating to the fluorescence of substances containing chromophoric groups are communicated, and a summary is given of the results obtained in the series of papers published by Stark on the connexion between fluorescence and absorption.

The new substances examined were acetone, methyl ethyl ketone, pyrotartaric acid, phorone, diacetyl, camphor, camphorquinone, methylfulven, potassium azodicarboxylate, azodicarbonamide, diethylketen, and diphenylketen. From the observations, the conclusion is drawn that if a substance possesses two absorption bands, one in the region of longer wave-lengths, the other in the remote ultraviolet region, it will fluoresce in the region of the first band when light is absorbed in the region of the second. The fact that the maxima of the intensity of the fluorescence and absorption do not coincide is due to the absorption which the fluorescent light undergoes in its passage through the absorbing medium. The two azo-compounds investigated are exceptions to this general statement, but this is supposed to be due to the absence of the radiation of the requisite very short wave-lengths from the aluminium light used in the experiments.

H. M. D.

Ultraviolet Fluorescence of Cyclic Compounds. HEINRICH RY and K. VON ENGELHARDT (*Ber.*, 1908, 41, 2988—2995. Compare Stark, Abstr., 1907, ii, 147; Stark and Meyer, *ibid.*, ii, 418).—The ultraviolet fluorescent, and the absorption, spectra of mono- and substituted benzene derivatives in ethyl-alcoholic solutions of 0.005 concentration have been measured in an apparatus very similar to that used by Stark; the results confirm this investigator's theory of the relation between fluorescence and selective absorption.

Toluene and propylbenzene are more strongly fluorescent than benzene, and the four bands which characterise the fluorescent spectrum of benzene have disappeared in the spectra of these two homologues. The auxochromic influence of the amino-group is accompanied by an auxofluoric influence, since aniline is more fluorescent than benzene; dimethylaniline, however, is less fluorescent than aniline, although the contrary might be expected from Kauffmann's

view that the dimethylamino-group is a stronger auxochrome than the amino-group. The hydroxyl and the methoxyl groups also act as auxochromes, phenol and anisole exhibiting a remarkably strong fluorescence, whilst the very feeble fluorescence of benzoic acid is markedly increased in the hydroxybenzoic acids, of which the ortho-compound fluoresces most strongly, even in the visible region, as is evident to the naked eye, when a quartz lamp and a quartz vessel are used. Although anisole is more fluorescent than phenol, the methoxybenzoic acids are less so than the hydroxy-acids; the fluorescence of *p*-methoxybenzoic acid cannot be detected with certainty. The introduction of a halogen into the benzene nucleus diminishes the fluorescence, the more so the greater the molecular weight; iodo-benzene does not fluoresce. Benzonitrile is remarkably fluorescent. Benzamide is non-fluorescent, showing that the amino-group only has auxochromic influence when situated in the nucleus.

The presence of a nitro-group often entirely prevents fluorescence even although *o*- and *p*-nitrophenols and *o*-nitroaniline exhibit marked selective absorption (according to Stark's theory, fluorescence is conditioned by the presence of bands in the absorption spectrum). The non-fluorescence may be attributable to quinonoid structure (compare Baly, Edwards, and Stewart, *Trans.*, 1906, 89, 514 Hantzsch, *Abstr.*, 1906, i, 353), since simple quinones never fluoresce. Neither pyridine nor ethyl collidinedicarboxylate exhibit fluorescence. Ethyl dihydrocollidinedicarboxylate, however, shows violet fluorescence in alcoholic solution, and also a very intense band; thus providing a good confirmation of Stark's theory. C. S.

Natural and Magnetic Rotation of the Plane of Polarisation in Crystals. WALDEMAR VOIGT and K. HONDA (*Physikal. Zeitsch.* 1908, 9, 585-590).—Measurements of the natural rotation of the plane of polarisation for a series of wave-lengths are recorded for *d*- and *l*-sodium chlorate and *d*-tartaric acid in the direction of the optic axis, and for sucrose in the direction of the two axes. The rotation induced by a strong magnetic field was measured for sodium chlorate, dolomite, apophyllite, apatite, and sucrose. In the case of sucrose, the values obtained for the two optic axes indicate that the magnitude of the rotation of the plane of polarisation is different in the two directions. H. M. D.

Relation between Rotatory Power and Chemical Constitution. II. D. HARDIN (*J. Chim. Phys.*, 1908, 6, 584-591. Compare this vol., ii, 470).—The theoretical deductions given in the previous paper have been further tested by application to certain derivatives of hexyl alcohol.

The atomic product for oxygen (the product of the distance between the oxygen and carbon atoms and the atomic weight of the former element) is approximately the same as that calculated from observation on the amyl derivatives, but is of opposite sign. The difference of sign has not been satisfactorily accounted for.

The theory also indicates that the distance between the centre of gravity of the benzene ring and the first carbon in the side-chain will be different according as the bonds between carbon and hydrogen are in the plane of the ring or perpendicular to it. The experimental data obtained with hexylbenzene support the latter view as to the structure of the benzene nucleus.

Active hexyl alcohol (γ -methylamyl alcohol) was prepared by saponification of Roman camomile oil with cold concentrated potassium hydroxide solution and repeated fractionation. The fraction boiling at 151—152°/758 mm. has D^{20}_D 0.8262, $[\alpha]^{20}_D$ + 8.77°, D^{30}_D 0.7812, $[\alpha]^{30}_D$ 7.53°, and D^{47}_D 0.7276, $[\alpha]^{47}_D$ 6.10°. The corresponding *bromide* has D^{19}_D 1.1852, $[\alpha]^{19}_D$ + 19.97°, D^{27}_D 1.1150, $[\alpha]^{27}_D$ 17.12°, and D^{42}_D 1.0319, $[\alpha]^{42}_D$ 13.97°. Active hexylbenzene (α -phenyl- γ -methylpentane) (compare Klages and Sautter, Abstr., 1904, i, 302), prepared by the action of sodium on a mixture of bromobenzene and hexyl bromide, has D^{19}_D 0.8521, $[\alpha]^{19}_D$ + 16.62°, D^{28}_D 0.7873, $[\alpha]^{28}_D$ 14.99°, and D^{38}_D 0.7396, $[\alpha]^{38}_D$ 13.66°.

G. S.

Optical Isomerides. I. IWAN OSTROMISLENSKY (*Ber.*, 1908, 41, 3035—3046).—The observations of Gernez on the separation of a *dl*-conglomerate by means of the addition of a fragment of a substance isomorphous with one of the components have been extended.

The addition of *l*-asparagine to a supersaturated solution of *dl*-sodium ammonium tartrate causes the deposition of the pure *d*-tartrate.

Both *d*-tartrate and *l*-asparagine are hemihedral forms belonging to the rhombic system, and are isomorphous.

An optically active salt can also be obtained by impregnating a supersaturated solution of *dl*-sodium ammonium tartrate with a crystal of an active modification of any other alkali tartrate; for example, Rochelle salt, potassium tartrate, sodium tartrate, potassium hydrogen tartrate. In all cases the salt which is deposited is dextrorotatory if the impregnating material is dextro, and laevo if this is laevorotatory. Inoculation with ordinary ammonium malate causes the deposition of *d*-sodium ammonium tartrate. Substances which are isodimorphous with the sodium ammonium tartrate can also be used for bringing about the separation. Thus a crystal of *d*-potassium ammonium tartrate (usually monoclinic) causes the deposition of *d*-sodium ammonium tartrate, and similarly with potassium tartrate.

It is not necessary that the material used for inoculation should be optically active; thus glycine added to a supersaturated solution of *dl*-asparagine causes the deposition of either *d*- or *l*-asparagine, and it is not possible in any given experiment to predict which of the two will separate from an inspection of the glycine crystals, although the same glycine crystal always acts in the same way. The conclusion is drawn that glycine belongs to the hemihedral class of rhombic prisms.

This method of deposition is suggested as a method for determining whether the given substance is a *dl*-conglomerate or a *r*-compound.

If the crystals deposited on impregnating with an isomorphous substance are found to be inactive, then the original solution contained

a racemic compound, but if active, then the original was an inactive conglomerate. J. J. S.

Chemical Action of Light. GIACOMO L. CIAMICIAN (*Bull. Soc. chim.*, 1908, [iv], 3, i—xxvii).—A lecture delivered before the French Chemical Society. E. H.

Chemical Action of Tropical Sunlight. GILBERT JOHN FOWLER (*Mem. Manchester Phil. Soc.*, 1908, 52, xiv, 1—20).—Observations are recorded on the rate of liberation of iodine from an acidified potassium iodide solution under the influence of light. The reaction is practically unaffected by temperature under the ordinary conditions of exposure. The rate of liberation of iodine from a given solution diminishes with the duration of the exposure; this is due to the absorption of the chemically-active rays by the iodine liberated previously. The initial rate of liberation diminishes with increasing dilution of the solution, but, on longer exposure, results in closer agreement are obtained, owing, no doubt, to the greater retarding effect of the iodine liberated from the stronger solutions.

Tables of data obtained by measurements made in Calcutta and on the Mediterranean and Arabian Seas are recorded. The highest record corresponds with an actinic effect which is forty-two times as great as the lowest record obtained on a bright December day in Manchester. No relationship appears to exist between the photochemical and the sun-stroke effect of sunlight.

Solutions of bleaching powder lose their available chlorine with great rapidity on exposure to tropical sunlight. The effect is much more marked in the case of dilute solutions than it is in strong solutions.

Silver chloride, sealed in a glass tube and darkened by exposure to light, becomes quite white again when kept for some time in the dark. In the presence of a globule of mercury, the original colour is not restored. These observations support the view that the darkening of silver chloride on exposure to light is a dissociation effect, and is not due to the formation of an oxychloride. H. M. D.

Photochemical Reactions. III. Decomposition of Ozone by Light. FRITZ WEIGERT (*Zeitsch. Electrochem.*, 1908, 14, 591—597).—The decomposition of ozone by light in presence of chlorine (this vol., ii, 5) is studied quantitatively. The light of a mercury quartz lamp is used, and the rate of decomposition measured by means of the change of pressure of the gas. There is no induction period, and decomposition stops when the light is cut off. The rate of decomposition is independent of the concentration of the ozone; it increases with the concentration of the chlorine, but not in proportion to it, and it is nearly proportional to the intensity of the light. By placing a vessel containing chlorine between the source of light and the ozone tube, a measurement of the quantity of light absorbed by the chlorine is obtained; in this way it is shown that the rate of decomposition of the ozone is proportional to the quantity of light absorbed by the chlorine mixed with it. A rise of temperature of 10° increases the rate of reaction 1.17 times

It is suggested that the light produces some intermediate product which is destroyed rapidly by the action of ozone; the rate of decomposition measured is then the rate of formation of this product, which is independent of the concentration of the ozone. T. E.

Catalytic Reactions and Photochemical Equilibria. BARTOLO L. VANZETTI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 285—288).—The author has carried out experiments to ascertain whether catalytic action necessitates immediate contact of the catalyst and the substance acted on, or whether the action can take place at a distance. When detonating gas is separated from platinum-black by a sheet of glass, the thickness of which is of the same order of magnitude as the wavelength of light, no combination occurs between the hydrogen and oxygen. But when the separating layer consists of an organic material, such as gelatin or a colloid, the hydrogen and oxygen combine slowly, but combination is found to be preceded by diffusion of the mixed gases through the membrane, as is shown by the observation that the diminution of the gas volume per unit of time is proportional to the concentration (pressure) of the gas mixture (compare Ernst, *Abstr.*, 1901, ii, 495).

Experiments with platinum and platinum-black placed at a distance of 0.5—1 mm. from rapid bromide plates show that both these substances exert a negative action on the plate, that is, they take away the darkening produced in the sensitive emulsion by the action of light (compare Piltchikow, *Abstr.*, 1906, ii, 414). This action is, however, quite different from that of light, and from the Russel effect produced by certain metals, in that it is only manifested at the surface of the photographic film and penetrates the latter only very slowly. This negative effect of platinum is especially marked in moist air, is greatly diminished in a cathode-ray vacuum, and is not exhibited in air in presence of phosphoric oxide. Experiments with other metals show that these may be arranged in the following series, in the first part of which the metals exhibit a diminishing positive effect on a photographic plate, whilst in the latter part an increasing negative effect is produced: cadmium, zinc, aluminium, lead, iron . . . copper, tin, silver, palladium, iridium, nickel, gold, platinum. If the condition in a silver bromide plate is represented as an equilibrium between the two reactions $3\text{AgBr} \rightleftharpoons \text{Ag}_2\text{Br} + \text{AgBr}_2$, the upper of which is produced by the action of light, and the lower by the action of darkness, the action of platinum at a distance is to accelerate the latter change. T. H. P.

Chemical Changes Produced by Different Kinds of Rays.
I. **Catalytical Reactions of Sunlight.** CARL NEUBERG (*Biochem. Zeitsch.*, 1908, 18, 305—320).—A large number of substances undergo chemical change when exposed in solution to sunlight in the presence of uranium salts. The following changes have been observed: Alcohols to aldehydes; poly-alcohols to hydroxy-aldehydes or ketones; acids to aldehydes or keto-compounds, sometimes with fewer carbon atoms than the original substance; monosaccharides to osones; disaccharides undergo inversion; polysaccharides and glucosides are

hydrolysed; amino-acids undergo deamidisation and lose carbon dioxide, yielding aldehydes with a smaller number of carbon atoms than the original substance; glycerides are partly hydrolysed; peptones and proteins are partly hydrolysed, and the hydrolysis products undergo the fuller changes noted above.

Amongst the products actually isolated by decomposition in sunlight in presence of uranium salts were: glycerose in the form of the osazone, m. p. 130—131°, from glycerol; glyoxal in the form of the *p*-nitrophenylosazone from tartaric acid; acetaldehyde (in the form of the *p*-nitrophenylhydrazone) from alanine, from which ammonia was also obtained; dextrose (in the form of the osazone) from inulin.

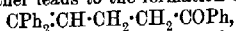
S. R. S.

Theory of Coloured Salts. HERMANN FECHT (*Ber.*, 1908, 41, 2983—2987).—The depth of the colour of many aromatic additive compounds appears to be conditioned by the magnitude of the difference of the electrical character of the two components; the more positive the one and the more negative the other, the deeper the colour. For example, quinone + phenol is red, quinone + sodium phenoxide is blue, and quinone + aniline in cold carbon disulphide yields a mass of dark violet crystals, which becomes colourless when the aniline is washed away. Again, diphenyldi-imine + benzidine gives a yellow solution, which becomes blue when the quinonoid component is made more negative by salt formation; the blue colour vanishes when the other component also enters into salt formation. Malachite-green becomes blue when chlorine is introduced into the phenyl group, and yellow when the halogen is attached to the still basic nitrogen atom. The effect on colour of the halogen atom or the hydroxyl or amino-group is not due to the influence of these *per se*, but depends on whether they increase or diminish the difference in the positive and negative characters of the components of the coloured substance.

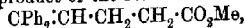
When chlorine is passed into a solution of di-iododiphenyl in chloroform, yellow crystals of the *dichloride*, $C_{12}H_8I_2Cl_2$, m. p. 146° (decomp.), are obtained. By heating the dichloride with benzidine in chloroform or carbon disulphide, a *substance*, $C_{24}H_{20}N_2Cl_2I_2$, is obtained in the form of a violet-blue powder, which sublimates and decomposes at 300°.

Equal molecular quantities of quinone and benzidine in chloroform yield a mass of bluish-violet needles with a red, metallic lustre, which melt indefinitely at 118°, resolidify, and decompose above 200°.

The reaction between methyl glutarate and excess of magnesium phenyl bromide in ether leads to the formation of the *ketone*,



m. p. 176°, which forms colourless crystals, and develops a bluish-green coloration with concentrated sulphuric acid. If the Grignard reagent is not in excess, the product of the reaction is the *ester*,



m. p. 120°.

C. S.

Rate of Decay of the Excited Radioactivity from the Atmosphere in Sydney. S. G. LUSBY and T. EWING (*J. Roy. Soc. New South Wales*, 1906, 40 158—160).—A copper wire, 10 metres

long, kept negatively charged at a potential of 25,000 volts, was after three hours' exposure rubbed with cotton wool moistened with ammonia. The cotton wool was incinerated, and the ashes tested for radioactivity; the time taken for the activity to fall to half value was on four days in December, 1905, found to be 41.5, 40.7, 41.9, and 39.5 minutes.

P. H.

Radioactivity of the Rocks in the Region Traversed by the Line to the Simplon. GINO GALLO (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 209—214).—The author has examined the radioactivity of various rocks excavated in the laying of the railway line to the Simplon. The limestone-gypsums and the rocks of the calcareous schist zone are either quite inactive or very feebly radioactive. With the orthogneisses, however, the chemical composition and coefficient of acidity of which indicate that they belong to the eruptive granitic magmas, moderately intense radioactivity is observed, the maximum value found being 43.2 volt-hours. The radioactivity is, in general, accompanied by the presence of certain minerals, such as titanite and zirconia. The pyrites minerals are also moderately radioactive, but the radioactivity cannot be definitely attributed to the metallic sulphides, and not to the gneissic gangue accompanying them. Borne (*Jahrb. Radioaktiv. Elektronik*, 1905) has shown that, of the springs issuing from these rocks, those which are richest in emanation have their origin in the gneiss or in the granitic rocks.

T. H. P.

Preliminary Observations on Radioactivity and the Occurrence of Radium in Australian Minerals. D. MAWSON and T. H. LABY (*J. Roy. Soc., New South Wales*, 1905, 38, 382—389).—A number of Australian minerals were examined for radioactivity by the ionisation produced in an air gap, the activity being compared with that of black oxide of uranium. Radium was tested for, and found in the case of two Australian minerals, but a gadolinite known to contain helium gave no radium emanation.

P. H.

Preparation of Radium from Pitchblende. HEINRICH PAWECK (*Zeitsch. Elektrochem.*, 1908, 14, 619—623).—A description of the process of manufacture of radium salts used at the radium works of the Austrian Government at St. Joachimsthal. The ores of Joachimsthal have been worked since 1518, first for silver, then for cobalt, nickel, arsenic, and bismuth, and more recently for uranium. In 1907 the extraction of radium from the residues from the uranium ore was begun. The raw ore is ground and washed to remove the lighter rock constituents, it is then roasted at 800° to remove sulphur, and finally heated again with sodium carbonate. Uranium, arsenic, antimony, tungsten, molybdenum, and vanadium are washed out in the form of sodium salts. From the residue, iron, aluminium, calcium, silicon, and lead are removed by boiling with sodium hydroxide and then with hydrochloric acid, very thorough washing following each operation. The residue contains barium and radium sulphates, which are converted into carbonates by repeated treatment with sodium carbonate and thorough washing, and then dissolved in hydrochloric

acid and reprecipitated by sulphuric acid. This treatment is repeated several times. The solution of the chlorides is finally evaporated to dryness, and extracted with concentrated hydrochloric acid, which leaves a residue consisting mainly of barium and radium chlorides, which is submitted to fractional crystallisation. Of the radioactivity of the original ore, 73.9% is found in the residue from the uranium extraction, and the remainder in the solid uranium salts produced; there is no loss in waste products.

T. R.

Quantitative Estimation of Radium Emanation in the Atmosphere. GEORGE C. ASHMAN (*Amer. J. Sci.*, 1908, [iv], 26, 119—120).—The author has determined the radioactivity of 200 litres of Chicago air taken from the ground level. The purified air was passed through a long copper spiral immersed in liquid air to condense the emanation, which was afterwards volatilised into an electro-scope and estimated. Precisely the same results were obtained in simultaneous estimations by this method, which is considered to be the best hitherto devised for the purpose.

The radium equivalent of the emanation per cubic metre of air was found to be 86, 58, and 45×10^{-12} gram in three experiments. Immediately after a thaw and heavy rain, the amount rose to 200×10^{-12} gram; under normal weather conditions with high barometer, the value 51×10^{-12} was found, whilst with a low barometer the amount was 131×10^{-12} .

The mean value, the equivalent of 1.0×10^{-10} gram of radium per cubic metre is 25% higher than the amount given by Eve. If the emanation has an appreciable vapour pressure at the temperature of liquid air, even the present estimate must be somewhat too low.

The active material had a half-period of decay of 3.5 days, indicating that it consisted only of radium emanation and that thorium emanation was absent.

R. J. C.

Amount of Radium Emanation in the Atmosphere. JOHN SATTERLY (*Phil. Mag.*, 1908, [vi], 16, 584—615).—The amount of radium emanation present in the atmosphere of Cambridge has been measured by two methods. In the first, the emanation in a known volume of air was absorbed by charcoal; in the second, it was condensed by means of liquid air. The electrical conductivity imparted to a given volume of air by the absorbed or condensed emanation was then compared with the conducting power brought about by the emanation emitted from a definite volume of a radium solution of known strength. The results of the two methods of measurement, which agree fairly well, show that the amount of radium emanation in the atmosphere varies very considerably. On the average, the quantity contained in a cubic metre of air is equal to the amount which would be in radioactive equilibrium with about 1×10^{-10} gram of radium. No connexion between the variation in the concentration of the emanation and the meteorological conditions could be detected. The observed values indicate that only a small proportion of the natural ionisation of the air is due to the presence of the radium emanation and its products.

H. M. D.

Amount of Radium Emanation in the Atmosphere near the Earth's Surface. A. S. EVE (*Phil. Mag.*, 1908, [vi], 16, 622—632. Compare this vol., ii, 7).—Experiments according to the method already described have been continued at intervals for almost a year. An error in the previous calculations is pointed out, and this is corrected in the evaluation of the new experimental data.

The average amount of emanation present in a cubic metre of the air of Montreal during the period of the experiments is that which would be in equilibrium with 60×10^{-12} gram of radium. The observed variation in the concentration of the emanation is such that the maximum is to the minimum as 7 is to 1. Changes of temperature have little or no influence on the concentration of the emanation, but it increases on the approach of a deep cyclone accompanied by heavy rain or a thaw of snow, and decreases with anticyclonic conditions when the weather is dry or very cold. H. M. D.

The Heat Developed by Radium. EGON VON SCHWEIDLER and V. F. HESS (*Monatsh.*, 1908, 29, 853—862).—A quantity of over 1 gram of radium chloride was used for the experiments. A differential calorimeter was used consisting of two gilt-copper vessels in a chamber at constant temperature. One vessel contained the radium, and the other was heated by means of an electric current. Two copper-nickel thermo-junctions were balanced against one another, the heating current being adjusted until the temperatures were equal.

The results indicate a heat development of 118.0 cal. per hour for 1 gram of metallic radium, with a possible error of 1.4%. This is in good agreement with the value, 117 cal., obtained by Ångström (Abstr., 1906, ii, 63). C. H. D.

Relation between Uranium and Radium. III. FREDERICK SODDY (*Phil. Mag.*, 1908, [vi], 16, 632—638).—A further examination of the carefully-purified uranium salts prepared in 1905–06 has been made to ascertain the rate of production of radium (compare Soddy and Mackenzie, Abstr., 1907, ii, 730). In one case, the growth of radium is not greater than 5×10^{-11} gram per kilo. of uranium in $2\frac{1}{2}$ years, and in the other, it is not greater than 1×10^{-11} per gram kilo. of uranium in $1\frac{1}{2}$ years.

An examination of the residues from the highly-purified uranium salts has shown that the amount of radium present has increased about seven times during a period of one year. This indicates that a radium-producing substance is present in commercial uranium salts. Further, since the residues had been purified by the barium sulphate method with the object of removing radium and any intermediate products in the transformation of uranium into radium, it is established that the barium sulphate purification process does not remove the whole of this radium-producing substance. H. M. D.

Radiation of Uranium X. MAX LEVIN (*Physikal. Zeitsch.*, 1908, 9, 655—657. Compare Abstr., 1907, ii, 836).—As the distance between the radiating substance and the ionisation chamber is diminished, the ionisation produced by uranium X slowly increases at

first, attains a maximum value, and then rapidly diminishes. If the active substance is covered with a thin layer of aluminium, there is no evidence of this maximum ionisation value. The following explanation of the observed maximum in the case of the uncovered substance is given. As the distance between the source and the ionisation chamber increases, the actual number of ions which are discharged increases. When this distance is considerable, the strength of the electric field is not sufficient to bring about the discharge of all the ions before reunion takes place. If the ionisation per centimetre were constant, this should lead to a constant limiting ionisation when the distance is sufficiently great, but the fact that the strongly ionised layer in the immediate neighbourhood of the active substance is gradually removed from the measuring chamber influences the ionisation in such a way that, although an increase takes place at first with increasing separation, this is more than compensated for by the second effect when the distance has attained a certain value.

H. M. D.

Actinium C, a New Short-lived Product of Actinium. OTTO HAHN and LISE MEITNER (*Physikal. Zeitsch.*, 1908, 9, 649-655).—

Measurements of the ionisation produced by the α - and β -radiation emitted by the induced activity of actinium indicate that this consists of three disintegration products: actinium A, B, and C. Actinium C was separated from A by the action of finely-divided metals and animal charcoal on a solution of the active deposit, and its period of decay was found to be 5.10 minutes. It emits no α -rays, but the β -rays previously attributed to actinium B are found to be due to actinium C. The α -rays given off by the active deposit are entirely due to actinium B, whilst actinium A only emits a very readily absorbed type of β -radiation. In agreement with Bronson's determination, the decay period of actinium B is found to be 2.15 minutes, and that of actinium A, thirty-six minutes. These two products can be separated by heating the active deposit for about thirty seconds in a blow-pipe flame; the separation is not, however, complete even when the temperature is raised to 2000°.

Equations are given which represent the change in the α - and β -activity of the active deposit in agreement with the experimental observations.

H. M. D.

Range of Activity and Absorption of α -Rays. Secondary Radiation Produced by α -Rays. E. ASCHKINASS (*Ann. Physik.*, 1908, [iv], 27, 377-390).—An apparatus is described by means of which the charge on α -particles from polonium could be measured after passing through aluminium foil in a high vacuum. A magnetic field was employed to remove electrons and secondary radiation.

As found by other workers, layers of aluminium foil up to 13.7 μ in thickness allow the whole of the α -particles to pass. At about 24 μ , the particles are completely arrested. It is probable that complete absorption occurs suddenly at a definite thickness of aluminium.

Experiments with the same apparatus in the absence of a magnetic field indicate that the negative radiation from polonium considerably exceeds the positive in quantity. The secondary radiation produced by the α -particles may also be studied by its means.

C. H. D.

Ionisation of Gases by α -Rays, and the Hypothesis of Initial Recombination of the Ions. M. MOULIN (*Chem. Zentr.*, 1908, ii, 474—475; from *Le Radium*, 1908, 5, 136—141. Compare Abstr., 1906, ii, 720).—The hypotheses of Bragg and Kleeman, and Langevin, as explanatory of the unsaturation of an electric current at low *E.M.F.* when passed through a gas ionised by α -rays, have been tested by the author. The evidence so far obtained appears to support Langevin's view, that the α -rays produce a larger number of ions, which, however, become arranged along the path of the α -rays.

J. V. E.

Quality of the Secondary Ionisation due to β -Rays. WILLIAM H. BRAGG and J. P. V. MADSEN (*Phil. Mag.*, 1908, [vi], 16, 692—697).—Experiments are described which show that the secondary rays reflected by different substances struck by the same bundle of primary rays are not uniform in quality, but vary from one substance to another. For the purpose of collecting all the secondary rays, the authors have made use of an ionisation chamber with a hemispherical wall.

It is found that the secondary rays returned by substances of low atomic weight contain a relatively large proportion of less penetrating rays. When the less penetrating portions of the primary beam are cut off, the emitted secondary rays are found to be more penetrating. On account of these facts, observations of secondary radiation cannot be accurately made when ionisation vessels of the ordinary type are employed.

H. M. D.

Attempts to Detect the Production of Helium from the Primary Radio-elements. FREDERICK SODDY (*Phil. Mag.*, 1908, [vi], 16, 513—530).—Experiments have been made to obtain a quantitative estimate of the amount of helium produced from uranium and thorium. By the process recently described (Abstr., 1907, ii, 251), it is possible to detect 2×10^{-10} gram of helium by observation of the D_3 line. In an experiment in which 350 grams of thorium in the form of nitrate were employed, helium to the extent of about 2×10^{-10} gram was detected after a period of accumulation of nearly seven months. This corresponds with a rate of production of 2×10^{-12} per year. The two estimated rates of production, on the assumption that the helium atom is identical with an α -particle, are, according to two different methods, 2×10^{-12} and 4.5×10^{-12} . It may be pointed out that the former value is almost eight times less than the value calculated three years ago.

H. M. D.

Rate of Production of Helium from Radium. Sir JAMES DEWAR (*Proc. Roy. Soc.*, 1908, 81, 4, 280—286).—The rate of production of helium from radium was determined directly by observing the growth of pressure in a McLeod gauge connected with a vessel containing 70 mg. of radium chloride. A small tube containing coconut charcoal was sealed on to the gauge; the tube was kept in liquid air during an experiment, and served to condense any gases, other than helium or hydrogen, which might be generated in, or leak

into, the apparatus. Before starting an experiment, the apparatus was exhausted, first with a mercury pump, then with a large bulb containing charcoal immersed in liquid air, which was sealed off after some hours, and, finally, to a pressure of 0.00005 mm. by cooling the small charcoal bulb.

The second and more satisfactory experiment lasted 1100 hours. At intervals, the radium was heated to low redness, causing an increase of pressure after cooling to room temperature, which remained steady, showing that part of the helium had been occluded by the radium. Direct experiment showed that the helium was not appreciably occluded by the charcoal. It was further shown by examination of the spectrum, and by two other independent methods, that the helium was not contaminated with hydrogen.

A mean line drawn through the observations with the radium heated, shows a steadily maintained helium increment of approximately 0.37 c.mm. per gram of radium per day, a result which agrees almost exactly with that calculated by Rutherford on the assumption that the α -particle is an atom of helium carrying two ionic charges. On the other hand, Ramsay and Cameron (*Trans.*, 1907, 91, 1274) have determined the rate of production of the emanation from radium, the ratio of the amount of helium to that of the emanation being found by them to be 3.18; their value is about eight times as great as that of the author. It is pointed out in a footnote that Rutherford's experimental determination of the rate of production of the emanation gives a value about one-tenth that obtained by Ramsay, but in good agreement with the present results, on the assumption that the ratio of helium to emanation is 3 : 1. G. S.

Accumulation of Helium in Geological Time. ROBERT J. STRUTT (*Proc. Roy. Soc.*, 1908, 81, A, 272—277).—The experiments were undertaken in order to obtain a comparison of the helium content of minerals with their geological age. It was found that phosphatic nodules and phosphatised bones, occurring in such strata that their relative age was well defined, possessed a radioactivity much higher than that of rocks, and that the activity was connected with the presence of uranium minerals.

The helium was liberated by dissolving the minerals in acids, and separated from other gases by special methods. The radium content of the minerals was determined in the usual way, and from the results the percentage of uranium oxide calculated by comparison with a standard uranium mineral.

The ratio of helium to uranium oxide is not strictly proportional to the age of the strata, but high ratios are not found in the younger strata, whereas they are common in the older ones. The absence of direct proportionality is ascribed to imperfect retention of the helium.

From the results, on the basis of Rutherford's estimate of the rate of production of helium from radium, an attempt is made to calculate the age of the strata. The following are provisional *minimum* values:

Phosphatic nodules of the crag, 225,000 years; of the upper green-

sand, 3,080,000 years; of the lower greensand, 3,950,000 years; and hematite overlying carboniferous limestone, 141,000,000 years.

G. S.

Helium in Saline Minerals, and its probable Connexion with Potassium. ROBERT J. STURTT (*Proc. Roy. Soc.*, 1908, 81, A, 278—279).—The ratio of helium to uranium oxide has been determined (as described in the previous abstract) for the Stassfurt minerals, sylvine, carnallite, kieserite, and rock salt, and a number of other minerals from the same source have been examined qualitatively. Only for sylvine and carnallite is the ratio so high that it cannot be accounted for by the uranium present, and it is suggested that potassium may be the source of the helium in these two minerals (compare Campbell and Wood, *Abstr.*, 1907, ii, 217; McLennan and Kennedy, this vol., ii, 750).

G. S.

Electrical Conductivity of Alloys and their Temperature-coefficients. ERNST RUDOLFI (*Physikal. Zeitsch.*, 1908, 9, 607—609).—Polemical against Guertler (compare this vol., ii, 557).

H. M. D.

Conductivity of Fused Salts. KURT ARNDT and ALBERT GESSLER (*Zeitsch. Elektrochem.*, 1908, 14, 662—665).—The conductivities of a number of salts are measured by the method described by Arndt (*Abstr.*, 1906, ii, 418). The results (in reciprocal ohms per cm.) are:

	450°	500°	550°	600°	650°	700°	750°	800°	850°	900°	950°	1000°	1050°	1100°
CaCl ₂	—	—	—	—	—	—	—	1·90	2·12	2·32	2·50	2·66	2·76	—
KCl	—	—	—	—	—	—	—	2·19	2·30	2·40	2·50	2·61	—	—
KBr	—	—	—	—	—	—	1·65	1·75	1·85	1·95	2·05	—	—	—
KI	—	—	—	—	—	1·39	1·51	1·64	—	—	—	—	—	—
NaBr	—	—	—	—	—	—	—	3·06	3·23	3·30	—	—	—	—
NaI	—	—	—	—	—	2·56	2·63	2·70	2·76	2·83	2·90	—	—	—
AgCl	—	—	4·34	4·48	4·62	4·76	—	4·98	—	5·14	—	—	—	—
AgBr	2·93	3·02	3·10	3·18	—	3·34	—	3·50	—	3·68	—	—	—	—
AgI	—	—	2·46	2·52	2·59	2·64	—	2·72	—	—	—	—	—	—
NaPO ₃	—	—	—	0·80	0·425	0·55	0·675	0·80	0·925	1·05	1·175	1·30	1·42	1·54
BaO ₃	—	—	—	—	—	—	—	0·7	1·2	2·1	3·3	4·6	× 10 ⁻²	—

The conductivity is in all cases a linear function of the temperature; for the alkali salts it is approximately proportional to the absolute temperature. The conductivities of mixtures of potassium and sodium chlorides and of calcium and strontium chlorides are very nearly the mean of those of the constituents.

T. E.

Density and Equivalent Conductivity of Some Fused Salts. KURT ARNDT and ALBERT GESSLER (*Zeitsch. Elektrochem.*, 1908, 14, 665—667).—The density is determined by means of a platinum sinker and Westphal balance. The results are (in grams per c.c.):

	850°	900°	950°	1000°	1050°
CaCl ₂	2·03	2·01	1·99	1·97	—
SrCl ₂	—	2·69	2·67	2·645	2·62
BaCl ₂	—	—	—	3·12	3·09

For mixtures of calcium and strontium chlorides, the densities found are slightly smaller than the mean :

	700°	800°	900°	1000°	1100°
NaPO_3	2.22	2.18	2.14	2.10	—
BaCl_2	—	—	1.520	1.508	1.496

Mixtures of sodium metaphosphate and boron trioxide were measured at 900°; very considerable contraction occurs. In the following table, α is the percentage of sodium metaphosphate :

α	50	25	10	5	1.0	0.5
Density ...	2.115	1.820	1.655	1.586	1.552	1.522

The equivalent conductivities are calculated by means of the figures in the preceding abstract.

T. E.

Limiting Conductivity of Some Electrolytes in Ethyl Alcohol. PAUL DUTOIT and H. RAPPEPORT (*J. Chim. Phys.*, 1908, 6, 545—551).—The alcohol used in the measurements had a specific conductivity of the order 10^{-7} reciprocal ohms. The values of the limiting molecular conductivity, μ_∞ , were obtained directly by measurements in very dilute solution (up to a dilution of 20,000 litres) and indirectly by extrapolation from measurements in more concentrated solutions. The values of μ_∞ at 18°, expressed in Siemens units, are 39.2, 39.1, 35.8, and 35.0 respectively for the iodides of potassium, sodium, lithium, and ammonium, and 41.1 and 38.0 for the thiocyanates of potassium and ammonium respectively. Ammonium bromide gives 36.4, and sodium acetate, 31.8.

Kohlrausch's law holds for the electrolytes examined within the limits of experimental error, but the dilution law does not apply.

The order of the speed of the ions is the same in water and in alcohol, but the ratio of the speeds in the two solvents is by no means constant, varying from 1.8—3.1.

G. S.

A Light Accumulator. R. GOLDSCHMIDT (*Bull. Soc. chim. Bely.*, 1908, 22, 317—327).—An accumulator in which the electrodes are zinc and oxide of nickel, and the electrolyte a concentrated solution of potassium carbonate, is described. The changes taking place on charging and discharging are represented by the upper and lower arrows respectively in the equation $\text{Ni}_2\text{O}_3 + 2\text{Zn} \rightleftharpoons \text{NiO} + 2\text{ZnO}$, the electrolyte taking no direct part in the reaction. The nickel peroxide electrodes are prepared by electrolysis of a solution of nickel silicofluoride containing a little cobalt silicofluoride, nickel plates being used as anode, and the nickel deposited on a sheet of iron covered with nickel serving as cathode. A constant *E.M.F.* of about 2.75 volts is employed. After seven to eight hours' electrolysis, it is necessary to add a little cobalt silicofluoride and free hydrofluosilicic acid to the electrolyte to prevent the formation of a green mass on the cathode. The plate thus prepared is washed and oxidised electrolytically in a solution of sodium carbonate.

The zinc electrodes are prepared by electrolysis of a strong solution

of zinc silicofluoride containing a little free acid, zinc plates being used as anode, and the zinc deposited on iron as cathode. It is remarkable that much better deposits of zinc are obtained after the electrolyte has been in use for some time.

The plates thus prepared are immersed in a 43% solution of potassium carbonate or a 25% solution of sodium carbonate to form an accumulator; the potassium salt is preferable. An accumulator of this type has a capacity of at least 60 ampere-hours per kilo. of positive electrode; with a current of 15–20 amperes, the yield is about 100 watts, and the mean *E.M.F.*, 1.57 volts. It has the further advantage that it can be charged with a very large current, up to 50 amperes per kilo. of positive electrode, without damage. G. S.

Formation of Gas in Discharge Tubes. RUDOLF VON HIRSCH (*Physikal. Zeitsch.*, 1908, 9, 603–606. Compare Hirsch and Soddy, this vol., ii, 12).—The author has investigated the gas which is evolved from aluminium electrodes containing sodium as an impurity when cathode rays are generated by means of an influence machine. About 50% of the gas is absorbed by glowing copper oxide, and consists of hydrogen. The remainder appears to be carbon dioxide, for the spectral bands of this gas become much stronger after the removal of the hydrogen. This mixture of gases differs from others previously examined in that the product p^2V is constant, p being the gas pressure and V the discharge potential. It has been suggested previously (*loc. cit.*) that the constancy of p^2V is characteristic of pure gases, and this has led to a detailed examination of mixtures of hydrogen and carbon dioxide.

The value of p^2V for pure carbon dioxide is about $1/22$ of that for hydrogen; the connexion between the value of the product and the molecular weight of the gas does not hold therefore in this case. For mixtures of the two gases, p^2V is not constant; the curve, which represents the deviations of the product from a constant value, exhibits maxima for mixtures containing a large excess of one or the other gas, and a minimum for which $p^2V = 4$.

Analysis of two mixtures giving this minimum value showed that approximately equal quantities of the two gases are present. This mixture is the one produced from the aluminium electrodes, the constancy of the proportions between the two gases being maintained automatically. The formation of the gases is due to the action of water and carbon dioxide on the sodium contained in the aluminium.

H. M. D.

Chemical or Physical Theory of Volatilisation (of Cathodes). FRANZ FISCHER and OTTO HÄHNEL (*Zeitsch. Elektrochem.*, 1908, 14, 677–681). VOLKMAR KOHLSCHÜTTER (*ibid.*, 681–683).—Fischer and Hähnel regard the volatilisation as a purely physical phenomenon; Kohlschütter upholds his chemical hypothesis (this vol., ii, 457, 653, 799, and 800).

T. E.

Volatilisation of Cathodes in Attenuated Gases. B. WALTER (*Zeitsch. Elektrochem.*, 1908, 14, 695).—In Fischer and Hähnel's experiments (this vol., ii, 653), the current passing in the argon

and hydrogen tubes is the same, but the energy consumed is greater in hydrogen; hence for equal quantities of energy the volatilisation would be greater in argon.

T. E.

Behaviour of Calcium and Sodium Amalgams as Electrodes in Solutions of Neutral Salts. HORACE G. BYERS (*J. Amer. Chem. Soc.*, 1908, 30, 1584—1586).—Le Blanc and Novotný (Abstr., 1907, ii, 22) found that the values of the equilibrium constant of the reaction between sodium carbonate and calcium hydroxide, obtained by direct determination, did not agree with those calculated from the solubilities of calcium hydroxide and calcium carbonate. An attempt has therefore been made to determine this constant by measuring the *E.M.F.* of the cell $\text{CaHg}-\text{CaCO}_3-\text{Na}_2\text{CO}_3 \mid \text{NaOH}-\text{Ca}(\text{OH})_2-\text{CaHg}$, K being calculated from the equation $\pi = RT/7 \times \ln K/a$. The value of π , however, could not be satisfactorily determined, owing to the rapid action of water on the calcium amalgam, but interesting results were obtained on the action of solutions of salts on the amalgam. Similar experiments were made with sodium amalgam and solutions of salts. The results are tabulated.

It has been found that when amalgams are treated with solutions of salts of alkali or alkaline-earth metals, replacement occurs, and the determination becomes that of the *E.M.F.* of an electrode consisting partly of the amalgam of the metal of the added solution. Replacements of this character in concentrated amalgams have been recorded previously.

E. G.

Anomaly of the Strong Univalent Electrolytes M. KATAYAMA (*J. Coll. Sci. Tokyo*, 1908, 25, vii, 1—42).—By combining the views of Nernst (Abstr., 1901, ii, 647) and Jahn (Abstr., 1900, ii, 522; 1901, ii, 491; 1902, ii, 597) with those of Storch (Abstr., 1896, ii, 288) and Bancroft (Abstr., 1900, ii, 186), the author has deduced the following empirical formula:

$$K = \frac{c_i^n}{c_n} \left(\frac{c_i}{c_n} \right)^{0.3 \frac{c_i^2}{c_n}}$$

that is, $\log K = n \log \frac{\lambda}{\lambda_\infty} c - \log \frac{\lambda_\infty}{\lambda} c - 0.3 \frac{\lambda^2 c}{(\lambda_\infty - \lambda) \lambda_\infty} \log \frac{\lambda}{\lambda_\infty - \lambda}$,

where K is a constant, c_i the concentration of the ions in gram-mols per litre, c_n the concentration of the neutral, undissociated molecules, $c_i + c_n = c$ the total concentration, n a constant, λ the equivalent conductivity for the concentration c , and λ_∞ the equivalent conductivity for infinite dilution. The values obtained with this formula are in close agreement with the observed conductivities for every kind of univalent electrolyte.

In conjunction with Nernst's theory of solutions (*loc. cit.*), the following assumptions were made: $\pi_1 = a_1 c_n c_i^s$; $\pi_2 = a_2 c_i \left(\frac{c_i}{c_n} \right)^{0.3 \frac{c_i^2}{c_n}}$; where π_1 is the vapour pressure of the neutral molecule, π_2 the vapour pressure of the cation or anion, and a_1 , a_2 , and s are constants. The

values for the *E.M.F.* calculated on the second assumption are in fair agreement with the observed values. The partition-coefficient values obtained by Rothmund and Drucker for picric acid (Abstr., 1904, ii, 231) are well explained with the aid of the first assumption. By combining the two, it is possible to calculate the lowering of the freezing point of an aqueous solution of a strong electrolyte, which as a rule is in agreement with the observed depression. W. H. G.

The Size of Molecules and Charge of the Electron. JEAN PERRIN (*Compt. rend.*, 1908, 147, 594—596).—The number (*N*) of molecules in a gram-molecule, the charge (*e*) of the electron, and the ratio (*a*) of the mean energy of a molecule to its absolute temperature are universal constants, all of which are known when the value of one is determined, since (1) in the electrolysis of a univalent salt 96550 coulombs are conveyed by *N* atoms, whence $Ne = 3 \times 10^9 \times 96550$ electrostatic units; (2) the kinetic energy of a gram-molecule is measured by $3RT$, so that $2Na = 3R = 3 \times 83 \cdot 2 \times 10^6$.

The values 3×10^{-10} (Townsend), $3 \cdot 4 \times 10^{-10}$ (J. J. Thomson), $6 \cdot 8 \times 10^{-10}$ (J. J. Thomson), and $3 \cdot 1 \times 10^{-10}$ (H. A. Wilson) have been found for *e*, and from them values varying from 43×10^{22} to 96×10^{22} derived for *N*. Planck has obtained the value $2 \cdot 02 \times 10^{-16}$ for *a* and deduced 61×10^{22} for *N*, but Lorentz from the same data finds $N = 77 \times 10^{22}$. The author has determined *N* by a method based on the assumption that equal particles distribute themselves in a dilute emulsion in the same manner as molecules of the same mass which obey the gas laws. This leads to the equation $2 \cdot 3 \log_{10} n_0/n = N/R[4/3\pi a^2 g(d-\sigma)h]$, in which *n* and *n*₀ signify the concentrations of the particles at levels separated by the distance *h*, *a* the radius of the particles, and (*d* - *σ*) the excess of their density over that of water. After finding that particles of very different size gave the same value for *N*, the value 71×10^{22} was obtained, and the values $4 \cdot 1 \times 10^{-10}$ and $1 \cdot 7 \times 10^{-16}$ thereby deduced for *e* and *a* respectively. From this value of *N*, the mass of the oxygen molecule is $0 \cdot 45 \times 10^{-22}$ gram, of the hydrogen atom $1 \cdot 40 \times 10^{-24}$ gram, and of the corpuscle $0 \cdot 75 \times 10^{-27}$ gram. E. H.

Magnetic Susceptibility of Solutions. P. PASCAL (*Compt. rend.*, 1908, 147, 242—244. Compare this vol., ii, 756).—The rule that a magnetic atom becomes more diamagnetic the more it enters into complex ions or colloidal molecules, and becomes removed from its characteristic chemical properties, has been verified in the cases of certain ferrous, cobalt, chromium, copper, and mercury salts. Ferropyrrophosphates, ferro-oxalates, and ferrometaphosphates are oxidised at rates which are in order of their magnetism. The decrease of magnetism on adding sodium carbonate to these salts is accompanied by a slower rate of oxidation. R. J. C.

Relation of Melting Point to the Surface of the Granules of a Solid Substance. P. N. PAWLOFF (*J. Russ. Phys. Chem. Soc.*, 1908, 40, 1022—1066).—From theoretical considerations, the author shows that the relation between the melting point, *T*, of a substance and the

radius, r , of its granules is expressed by the following differential equation:

$$dT/dr = [A(v_2 - v_1)p \cdot 2k/r^2 \cdot dR\sigma] / [\rho + A(v_2 - v_1)p \cdot 2k/r \cdot dR\sigma T],$$

where A is a constant independent of the magnitude of the granules for any given temperature, and is given by the expression: $\log Ap = 2k/rdR\sigma T$; k is the surface tension at the separation of the solid from vapour; r the radius of the spherical granules; σ the specific volume of the vapour; ρ the specific heat of liquefaction; v_2 and v_1 the volumes of unit masses of the liquid and solid respectively at the melting point.

Granules of commercial salol having a magnitude of 8μ melt 3.5° lower than those 32μ in size. After one recrystallisation, granules of 6μ — 40μ melt within an interval of 1.5° . Dust having dimensions less than 2μ melts, in the case of salol 7° , with antipyrine 5 — 7° , and with phenacetin 4° , lower than large granules of diameter 0.5 — 2 mm.

T. H. P.

Apparatus for Determining the Freezing Point of Mixtures.

Otto SCHEUER (*J. Chim. Phys.*, 1908, 6, 620—624).—Two forms of apparatus suitable for determining the freezing point of mixtures are described. The first is a modified Beckmann apparatus. The essential feature of the second is that the stirring is concentric, and is effected by means of the thermometer, to the lower part of which two strips are attached for this purpose.

G. S.

Negative Adsorption. REGINALD O. HERZOG (*Zeitsch. physikal. Chem.*, 1908, 57, 315—316).—Tezner and Roske state (this vol., ii, 810) that negative adsorption does not occur. This, however, is only true when the adsorption of a single substance is dealt with, and instances are given where, under other conditions, the phenomenon does take place.

W. D. H.

The Fusion Curves of the System Naphthalene-Phenol.

T. YAMAMOTO (*J. Coll. Sci. Tōkyō*, 1908, 25, xi, 1—23).—The freezing-point curve of mixtures of naphthalene and phenol was determined by the cryoscopic method, a correction being made for supercooling. The eutectic point lies at 29.7° and 15.7 mol. % naphthalene. The form of the phenol curve is explained as being due to the association of phenol in the liquid state, represented by the equilibrium $3C_6H_5O \rightleftharpoons (C_6H_5O)_3$. The equilibrium constant of this association is expressed as a function of temperature, the heat of association being also determined.

C. H. D.

The Fusion Surfaces of the System Naphthalene-Chlorobenzene-Phenol, and the Molecular Association of Phenol. H. HIROBE (*J. Coll. Sci. Tōkyō*, 1908, 25, xii, 1—50. Compare preceding abstract).—The fusion surfaces (freezing-point surfaces) of naphthalene and phenol in the ternary system have been determined, an accurate cryoscopic method being employed. The two surfaces are separated by a eutectic curve, the projection of which on the plane of composition is nearly a straight line. The form of the surfaces is

consistent with the assumption that naphthalene and chlorobenzene are non-associated, and that phenol is partly associated to triple molecules. The latter conclusion was supported by determinations of the partition-coefficient of phenol between chlorobenzene and an aqueous solution of sodium chloride and sodium sulphate. C. H. D.

Arrangement for Liquefying and Distilling Easily Condensable Gases and Low-boiling Liquids. AUREL VON BARTAL (*Chem. Zeit.*, 1908, 32, 891).—A U-shaped form of receiver which can be completely immersed in a freezing mixture contained in a vessel consisting of two concentric cylinders between which there is a layer of asbestos; in this way, a very effective condensation is ensured. When removed from the freezing mixture, the apparatus can be employed as a distillation flask by gently warming it; the distillate may be condensed in a second similar apparatus cooled in a freezing mixture. P. H.

Practical Studies in Vacuum Sublimation. RICHARD KEMPF (*J. pr. Chem.*, 1908, [ii], 78, 201—259).—The employment of sublimation in a vacuum as a means of purification entails a smaller loss of substance than does recrystallisation, whilst, on the other hand, in consequence of the slower volatilisation, a more complete separation of a mixture is secured than by fractional distillation. The author describes an arrangement for sublimation experiments suitable for use in a laboratory, consisting of a glass tube on to one end of which is ground a glass cap with a tap and nozzle for connecting with the pump, and on to the other a bent pear-shaped glass bulb. This bulb, containing the substance to be sublimed, is heated in a suitable air-oven; the straight tube projecting through a hole in the side of the oven serves as the receiver for the sublimate. The phenomena of sublimation are discussed, and details given of experiments with fifty-three inorganic and organic compounds. G. Y.

Heat of Combination of Acidic Oxides with Sodium Oxide and Heat of Oxidation of Chromium. WILLIAM G. MIXTER (*Amer. J. Sci.*, 1908, [iv], 28, 125—137. Compare Abstr., 1907, ii, 738).—The heats of formation of sodium orthoborate, aluminate, chromate, and tungstate have been determined by firing mixtures of sodium peroxide with the metals or with metallic oxides in presence of carbon. Incidentally, the heats of formation of chromium sesquioxide and trioxide were calculated. Since the salts in question are largely hydrolysed by water, previous determinations by Thomsen, Berthelot, and others which referred to solutions are regarded as less trustworthy than those obtained by the sodium peroxide method. The heat of formation of sodium sulphate, on the other hand, is found to be the same by both methods. The principal results obtained were:

$3\text{Na}_2\text{O} + \text{B}_2\text{O}_3 = 2\text{Na}_3\text{BO}_3$	104,200 cal.
$\text{Na}_2\text{O} + \text{Al}_2\text{O}_3(\text{amorphous}) = 2\text{NaAlO}_2 +$	40,000 "
$\text{Na}_2\text{O} + \text{CrO}_3 = \text{Na}_2\text{CrO}_4$	77,000 "
$\text{Na}_2\text{O} + \text{WO}_3 = \text{Na}_2\text{WO}_4$	94,700 "
$\text{Cr} + 3\text{O} = \text{CrO}_3$	140,000 "
$2\text{Cr} + 3\text{O} = \text{Cr}_2\text{O}_3(\text{amorphous})$	243,800 "

The heats of transformation of aluminium and chromium oxide from the amorphous to the crystalline state were also determined.

During the combustions which were made in a silver bomb, or a silver cup suspended in a steel bomb, a small amount of oxygen was always liberated, for which a correction was necessary. R. J. C.

Viscosity of Certain Metals and its Variation with Temperature. CHARLES E. GUYE and S. MINTZ (*Arch. sci. phys. nat.*, 1908, 23, 136—166, 263—278).—From observations on the rate of decrease of the amplitude of vibration of loaded wires when twisted, the authors have compared the viscosity of platinum, silver, gold, copper, and steel at different temperatures. To eliminate the damping effect of the air, the vibrating wire was enclosed in an apparatus which could be exhausted.

The viscosity increases with the temperature in the case of platinum, silver, gold, and copper, and the curves which express the relationship between the two factors are of the same type. In the case of steel, the viscosity increases up to about 100°, then falls, and passes through a minimum value at about 200°, above which it increases very rapidly. This phenomenon can be observed both with rising and falling temperatures, indicating that it is due to reversible changes.

The influence of temperature on the viscosity is diminished by previous annealing; it increases when the load on the wire is increased. H. M. D.

Viscosity of Dilute Alcoholic Solutions. TOSHIO HIRATA (*J. Coll. Sci. Tōkyō*, 1908, 25, v, 1—15).—Arrhenius has shown (Abstr., 1880, 336) that the viscosity of an aqueous solution of any two indifferent substances may be expressed by the formula $\eta = A^x B^y$, where A and B are two constants characteristic of the two solutes at a constant temperature, x and y being their respective volume percentages. With the object of ascertaining whether ethyl-alcoholic solutions follow the same law, the viscosities of ethyl-alcoholic solutions of various liquids, chiefly organic, have been measured. It is found that the formula can be applied to the solutions of all the substances examined, the error being less than 1% for solutions containing less than 10% by volume of the dissolved substance. Water, however, forms an exception; in this case, the formula can be applied only to very dilute solutions. When the concentration exceeds 3% by volume, the error is greater than 1%. W. H. G.

Viscosity and Ionic Volume. FREDERICK H. GETMAN (*J. Chim. Phys.*, 1908, 6, 577—583. Compare this vol., ii, 464).—Jones and Veazey (Abstr., 1907, ii, 438) have pointed out that the salts which diminish the viscosity of water have a large ionic volume, and there is evidence that for salts with the same anion the diminution of viscosity of the solvent is proportional to the atomic volume of the cation. In the present paper, this relationship is further illustrated by application to a number of nitrates and chlorides.

In order to show the relationship more clearly, the viscosity of solutions of the same salts is compared, when the degree of dissociation

tion in each case is 75%. Under these circumstances, the viscosity, η , is represented by the formula :

$$10^4 \times \eta = 89 \pm 0.0242C_m$$

where C_v is the atomic volume of the cation, and 89 represents the viscosity of water at 25°, there being good agreement between found and calculated values. On the other hand, there is no apparent connexion between the viscosity and the sum of the volumes of the ions and unionised molecules. G. S.

Changes in Internal Friction due to Protein Degradation. KARL SCHÖRR (*Biochem. Zeitsch.*, 1908, 13, 173—176).—Experiments were carried out with dialysed salt-free serum, which was treated with sodium hydroxide in various dilutions. An Ostwald viscosimeter was used for the measurements.

On treatment with the alkali, there is at first an increase in the internal friction, which is followed by a steady fall. A similar phenomenon is brought about by hydrochloric acid ; in the latter case, however, the fall is followed by a second rise, which is accompanied by a gradual separation of a precipitate from the liquid, which is finally sufficient to impede entirely the flow of liquid through the viscosity tube. S. B. S.

Diffusion of Gaseous Ions. ÉDOUARD SALLES (*Compt. rend.*, 1908, 147, 627—629).—The author has measured the coefficient of diffusion of gaseous ions towards the walls of narrow metallic containing tubes. The coefficient for positive ions is 0.031, and for negative ions 0.041, the value being unaffected by interchanging tubes of German silver, brass, and steel. Contrary to J. J. Thomson's supposition, the nature of the metal has no specific influence on the rate of diffusion of gaseous ions towards it. R. J. C.

Osmotic Pressure of Concentrated Solutions of Non-electrolytes. OTTO SACKUR (*Chem. Zentr.*, 1908, ii, 473 ; from *Jahrb. Schlesisch. Ges. Vaterl. Kultur*, 1908, 86, 1—14).—According to the experiments of Morse and Frazer, aqueous sucrose solutions above 20° exert an osmotic pressure equal to the corresponding gas pressure, when the concentration is defined as the number of molecules of the dissolved substance in a volume of the solvent (*Abstr.*, 1907, ii, 744). At lower temperatures, this agreement is not observed. If, however, the concentration is regarded as being expressed by the number of molecules of dissolved substance in a volume of the solution, the equation $p(v-b)=RT$ is applicable for all the observations with sucrose and dextrose. Contrary to the gas law, b , in the above equation, varies considerably with alteration of temperature ; for sucrose, it decreases from 0.31 (per litre) to 0.20 when the temperature rises from 0° to 20°, and for dextrose, from 0.16 to 0.093 under a like change of temperature. The author suggests that this is due to the hydration of the dissolved substance decreasing with rise of temperature. J. V. E.

Polymerisation and Dissociation of Molecules in the Liquid State. GEORGE G. LONGINESCU (*J. Chim. Phys.*, 1908, 6, 552—566. Compare *Abstr.*, 1905, ii, 79).—With reference to the relation between

the boiling point and molecular weight, Walker has pointed out that the formula $T/\sqrt{m}=37$ (m =molecular weight; T =boiling point on the absolute scale) holds more particularly for a number of hydrocarbons, but is not generally applicable. The author now shows that the modified expression $T/\sqrt{m+(1000\sqrt{m})/T}$ is about 64 for all the normal substances examined, and varies from 65 for slightly polymerised substances to 99 for water.

The above equation has two roots, $\sqrt{m_1}=T/37$ and $\sqrt{m_2}=T/27$. The ratio T/\sqrt{m} is termed x_1 , and $1000\sqrt{m}/T$ is termed x_2 . It is only for certain hydrocarbons and some other substances that $x_1=37$ and $x_2=27$; for the majority of substances, x_1 is less than 37, and x_2 greater than 27. This is taken to mean that, in the liquid state, the molecules of most substances are partly polymerised and partly dissociated, the mean of the weights of the molecules being the theoretical molecular weight, and the views of other observers are adduced in support of this opinion.

The "factor of dissociation," $K_1=(x_1/37)^2$, and the "factor of polymerisation," $K_2=1.88K_1$, are given in tabular form for all the liquids considered.

G. S.

The Chemical Theory of Solutions. I. K. IKEDA (*J. Coll. Sci. Tôkyô*, 1908, 25, x, 1—80).—An ideal solution is defined as a homogeneous liquid mixture, of which the volume and energy are the sum of the volumes and energies respectively of the components in the liquid state under the same pressure and at the same temperature. An attempt is made to explain all deviations from the properties of an ideal solution by assuming reversible chemical reactions within the solution and applying the law of mass action. The treatment is mathematical, and the results are applied to concrete cases.

C. H. D.

Hardness of Solid Solutions of Metals and of Definite Chemical Compounds. NICOLAI S. KURNAKOFF and S. F. SCHEMTSCHUSCHNY (*Zeitsch. anorg. Chem.*, 1908, 60, 1—37; *J. Russ. Phys. Chem. Soc.*, 1908, 40, 1067—1104).—The degree of hardness of a number of binary alloys, the structure of which is known, is plotted against the composition, and in the majority of cases the electrical conductivity is represented on the same diagram. The data for the bronzes and brasses and certain other alloys are due to previous observers, and the authors have themselves determined the hardness of the binary alloys copper-nickel, gold-copper, and gold-silver. It is shown that practically the same results are obtained whether the hardness is determined by the sclerometer (ruling lines on a prepared surface of the alloy), by the resistance to penetration, or by other methods.

The more important results of the investigation are as follows: (1) the formation of mixed crystals is accompanied by an increase in hardness; (2) the hardness of an unbroken series of mixed crystals can be represented by a continuous curve showing a maximum; (3) in the latter case, the maximum of the curve of hardness coincides with the minimum of the curve of electrical conductivity. (4) At

points corresponding with the extremities of the series of mixed crystals (when the miscibility of the metals is incomplete), there are breaks in the curve of hardness.

Experiments have also been made on the pressures required to force certain plastic binary mixtures in a thin stream through a small opening, and the pressures just sufficient for this purpose are plotted against the composition. These limiting pressures are roughly proportional to the hardness of the alloys. The curves for the isomorphous mixtures of *p*-dichloro- and *p*-dibromo-benzenes and of silver chloride and bromide show maxima, but that for zinc-tin alloys shows no maximum, corresponding with the fact that these metals have little or no tendency to form solid solutions.

Finally, when the metals form a chemical compound, the latter may be harder or softer than either of the components, and the curve obtained by plotting hardness against composition may show either a maximum or minimum. A number of examples illustrating this case are cited. G. S.

Causes Modifying the Dominant Faces of Crystals: Solid Solutions. PAUL GAUBERT (*Compt. rend.*, 1908, 147, 632—635. Compare Abstr., 1906, ii, 343).—Foreign matter is distributed between crystals and their mother liquor in a definite ratio. The crystalline habit may be modified, not only by crystallisable foreign matter, but by liquid molecules, and even by the mother liquor itself.

Crystals of phthalic acid from aqueous solutions are modified in the same sense by methyl, ethyl, and propyl alcohols, glycerol or aldehyde in small quantities. The minute amount of alcohol in the crystals augments as the proportion of alcohol in the mother liquor increases, but the alcohol is not merely in the state of occluded mother liquor. Methylene-blue, which favours a different crystalline habit in phthalic acid, also hinders the entrance of alcohol into the growing crystals. R. J. C.

Ultra-microscopic Observations. J. REISSIG (*Ann. Physik*, 1908, [iv], 27, 186—212).—The colloidal solutions of brown, tellurium, selenium, and silver, and of red and blue gold, prepared by Paal (compare Abstr., 1902, ii, 500, 508; 1905, ii, 158) have been examined by means of the ultra-microscope. These solutions contain the sodium salts of protalbic and lysalbic acids (compare Abstr., 1902, i, 653), which act as protective colloids. From the number of particles contained in a given volume, the linear dimensions of the particles of the colloidal elements are calculated. It is found that the number of observable particles is not proportional to the concentration of the dissolved colloid, for all the particles can only be seen in dilute solutions. The number of particles seen through the ultra-microscope is, for a given solution, proportional to the intensity of the source of light used.

The ultra-microscope has also been employed to examine the behaviour of colloidal gold solutions on the addition of hydrochloric acid. The slowness with which flocculation takes place is supposed

to represent the stability of the solution, and for both red and blue solutions, one maximum and two minimum positions of stability have been identified. The position of maximum stability corresponds with the isoelectric point of the solution. In dilute solutions the stability of the colloidal gold is greater than it is in concentrated. The rate at which a given quantity of hydrochloric acid causes flocculation to take place depends on the concentration of the acid, the rate decreasing as the concentration of the acid decreases.

H. M. D.

The Dynamic Theory of a Reversible Chemical Reaction. II. ERNST COHEN and TH. STRENGERS (*Chem. Weekblad*, 1908, 5, 719—721. Compare this vol., ii, 824).—Polemical. A reply to Smits and Wibaut.

A. J. W.

The Dynamic Theory of a Reversible Chemical Reaction. ANDREAS SMITS and J. P. WIBAUT (*Chem. Weekblad*, 1908, 5, 771—772).—Polemical. A reply to Cohen and Strengers (this vol., ii, 824).

A. J. W.

Lecture Experiment to Demonstrate the Law of Mass Action. EMIL ABEL (*Zeitsch. Elektrochem.*, 1908, 14, 630—631).—The diminution of the concentration of the hydrogen ions in a solution of acetic acid by sodium acetate is shown by means of the reaction between hydriodic acid and hydrogen peroxide. In acid solutions, iodine is liberated; in almost neutral solutions, oxygen is given off. The difference between a solution of acetic acid, potassium iodide, and hydrogen peroxide, on the one hand, and the same solution with the addition of sodium acetate, on the other, is very striking (compare this vol., ii, 939).

T. E.

Application of the Kinetic Theory to the Metals. G. REBOUL (*Chem. Zentr.*, 1908, ii, 473; from *Le Radium*, 1908, 5, 129—136).—It is assumed that in metals there are free negative electrons and positive atom ions. The free electrons must, then, have a definite tendency to pass from the metal into the surroundings. It therefore follows that at the perimeter there must be a double film metal/gas, and also a potential difference, as in the Nernst theory for the perimeter metal/solution. The equilibrium between the electrons that leave the metal and the electrical energy is influenced by a change of temperature. In this is found a simple explanation of the phenomena of electric capillarity, thermal electricity, and electron rays when substances are strongly heated.

J. V. E.

Velocity of Reaction. JOHANNES J. VAN LAAR (*Chem. Weekblad*, 1908, 5, 747—764).—A theoretical paper containing a continuation of the author's statement of his new thermodynamic theory (this vol., ii, 824).

A. J. W.

Equilibrium of Two Substances in a Mixed Binary Solvent. E. D. VOLCHONSKY (*J. Russ. Phys. Chem. Soc.*, 1908, 40, 941—993).—A theoretical paper, in which the author considers: (1) The coefficient of distribution in a homogeneous system.

(2) Le Chatelier's principle of the opposition of action and reaction, and the conditions of existence of inverse systems. (3) Equilibrium of heterogeneous systems. (4) The energy relations between the members of a system. (5) The influence of temperature on the equilibrium of the system. (6) The classification of the reactions effecting the equilibrium of the system.

T. H. P.

Equilibria in Quaternary Systems. FRANS A. H. SCHREINERMAKERS (*Proc. K. Akad. Wetensch. Amsterdam*, 1908, 11, 138—142. Compare this vol., ii, 571).—The system copper sulphate, ammonium sulphate, lithium sulphate, and water has been studied at 30°. In addition to the three sulphates named, there are two solid compounds involved in the equilibria, namely, $\text{CuSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ and $\text{Li}_2\text{SO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$. The equilibria are represented with the aid of a tetrahedron, all saturation lines and surfaces being projected perpendicularly on one of the side planes of the tetrahedron. J. C. P.

Kinetics of Successive Reactions. Reduction of Chromic Acid by Oxalic Acid. K. JABŁCZYŃSKI (*Zeitsch. anorg. Chem.*, 1908, 60, 38—49.* Compare Luther and Rutter, *Abstr.*, 1907, ii, 555).—The rate of reaction between chromic acid and excess of oxalic acid in dilute solution has been measured at 15°, the course of the reaction being followed by estimating the concentration of chromic acid in a portion of the solution from time to time with iodide and thiosulphate. The velocity is proportional to the concentration of the chromic acid when different initial concentrations of the latter are used, but the constants calculated according to the unimolecular formula increase considerably during the reaction. Excess of chromic salt does not influence the rate of the reaction.

On the assumption that the reaction takes place in the three stages $\text{Cr}^{\text{VI}} \rightarrow \text{Cr}^{\text{V}}$ (1), $\text{Cr}^{\text{V}} \rightarrow \text{Cr}^{\text{IV}}$ (2), $\text{Cr}^{\text{IV}} \rightarrow \text{Cr}^{\text{III}}$ (3), and that all three reactions are unimolecular, an equation is deduced from which, by an approximation method, the following values are obtained for the constants of the three successive reactions $k_1 = 0.0344$, $k_2 = 0.1322$, $k_3 = 0.0238$. Other measurements were made in which the conditions were varied considerably, but, although the ratio of the constants calculated from these observations varied somewhat, the order remained the same.

The above conclusion, that the reaction $\text{Cr}^{\text{V}} \rightarrow \text{Cr}^{\text{I}}$ is very rapid and that the other reaction, $\text{Cr}^{\text{IV}} \rightarrow \text{Cr}^{\text{III}}$, is comparatively slow, is in accordance with what is known of the behaviour of quinquivalent and quadrivalent chromium salts.

G. S.

Formation of Salts and Basicity of Acids. GIUSEPPE BRUNI (*Zeitsch. Elektrochem.*, 1908, 14, 701—706).—Measurements are made of the conductivity of an acid and of mixtures of the acid and its potassium salt. In each series of measurements, the total concentration of the acid is kept constant, the series extending from pure acid at one end to pure potassium salt at the other. Acetic, formic, monochloroacetic, cyanoacetic, and di- and tri-chloroacetic acids are used; they form a series of gradually increasing strength. Each acid is used in eleven different concentrations from N to $N/1024$.

* and *Bull. Acad. Sci. Cracow*, 1908, 603—620.

The results are given in the form of tables and curves. For acetic acid of normal strength, the molecular conductivity increases as the neutralisation proceeds; in weaker solutions a minimum occurs near the acetic acid end of the curve. This minimum becomes more pronounced, and is found nearer the normal salt end of the curve, with formic acid; with the stronger acids the conductivity of the acid is greater than that of its potassium salt (except in the stronger solutions), and the minimum becomes less marked as the acid used is stronger, until with trichloroacetic acid it disappears altogether, the curves being straight lines. The theoretical discussion of the results is reserved for a later paper.

T. E.

Reduction of Solutions of Potassium Permanganate Acidified with Sulphuric Acid in the Presence of Mineral Salts. T. WARYNSKI and P. TCHEICHVILI (*J. Chim. Phys.*, 1908, **6**, 567—576).—The experiments were made with approximately 1/100 molar solutions of potassium permanganate and 1/30 molar sulphuric acid, and varying concentrations of salts. In some cases observations extended over four months. After the experiment, the amount of change was determined by titration with ferrous sulphate.

Most of the salts used as catalysts exerted an accelerating action in the dark, but a retarding action in daylight. Also, in green and blue light most salts retarded the action, but in orange light a slight acceleration was observed; the retarding effect, therefore, probably depends on the actinic rays.

Rise of temperature also affects the catalytic power of salts on the reaction; it increases the activity of the sulphates of iron, chromium, and aluminium, but retards the action of cadmium sulphate. The activity of certain catalysts, such as ferric alum, increases with the concentration.

G. S.

Esterification of Trichloroacetic Acid. ANTON KAILAN (*Monatsh.*, 1908, **29**, 799—844. Compare this vol., ii, 28).—The esterification of mandelic acid is not retarded by the presence of water up to 4 gram-mols. per litre. The influence of water on the esterification of phenylpropionic acid is much less when trichloroacetic acid is used as a catalyst than when hydrogen chloride is employed.

The relation between the velocity constant and the concentrations of the water and hydrogen chloride in the esterification of trichloroacetic acid is expressed by the equation: $1/k = 6.554 + 4.850/c + 0.0985/c^2 + (-40.15 + 35.39/c - 0.9550/c^2)w + (-16.90 + 12.98/c - 1.259/c^2)w^2$. The ratio of the velocity constants with hydrogen chloride and trichloroacetic acid respectively as catalysts is for phenylpropionic acid four times as great as for trichloroacetic acid. The presence of ethyl trichloroacetate does not retard the esterification. The ester is very little hydrolysed by the catalysts in nearly dry alcohol, but considerably in presence of water. The results are not in accordance with the theory of catalysis by hydrogen ions.

C. H. D.

Method of Calculating Atomic Weights. LOUIS DUBERUL (*Compt. rend.*, 1908, **147**, 629—632).—The method introduced by Hinrichs (*Abstr.*, 1907, ii, 945) of calculating the atomic weights of

all the elements present in a single reaction depends on a graphic construction, and is thus limited to reactions in which not more than three elements participate. The author develops Hinrichs' theorem analytically, obtaining equations whereby true atomic weights can be calculated from any reaction, however complex. The method consists in finding the minimum deviations from round number atomic weights which will account for experimental values within their limits of error. The true deviations are then to be obtained by averaging results obtained by different experimenters and methods.

R. J. C.

Connexion between Residual Affinity and Additivity. WALTER PETERS (*Ber.*, 1908, 41, 3175—3186).—Although the compounds of ammonia with the salts of heavy metals have been fully examined, the compounds with the salts of more strongly electro-positive metals have not been so closely examined, owing to their high dissociation pressure. The investigation has been made by means of Ley and Wiegner's apparatus (*Abstr.*, 1905, i, 749), as the formation of the additive ammonia compound occurs in an atmosphere of ammonia, and it also allows of the effect of temperature being determined.

Well-defined increase in the number of ammonia molecules absorbed by lowering the temperature was only given by silver chloride, platinum-chloride, bromide, and platinocyanide; in the other cases, ammonia was absorbed, and liquids were obtained containing a large percentage of ammonia.

The table summarises the results obtained at the ordinary temperature, I giving the number of ammonia mols. absorbed, and II the number retained after evacuation:

	I.	II.		I.	II.
Cobalt cyanide	2	0	Potassium aurithiocyanate var.	4	
Cobalt thiocyanate	6	2	Platinous chloride	5	4
Nickel thiocyanate	6	3	Silver platinochloride	6	3
Nickel dithionate	6	4	Potassium platinocyanide ..	1	0
Mercuric thiocyanate	4	1	Silver platinocyanide	4	2
Mercuric acetate	4	2	Cobalt platinothiocyande ..	6	2
Mercuric propionate	4	2	Potassium platinothiocyane		
Silver chloride	1.5	0	ate	2	2
Silver bromide	1.5	0	Silver platinothiocyande ..	6	4
Silver iodide	0.5	0	Sodium platinichloride ..	6	5
Silver cyanate	1	0	Silver platinichloride	8	4
Silver thiocyanate	2	0	Potassium platinithiocyan-		
Potassium aurichloride ..	3	0	ate	12	6
Potassium aurothiocyanate	5	2	Silver platinithiocyanate...	14	6

The platinic salts, in general, absorb more ammonia than the platinous, and the metals, gold or platinum, more than silver or mercury.

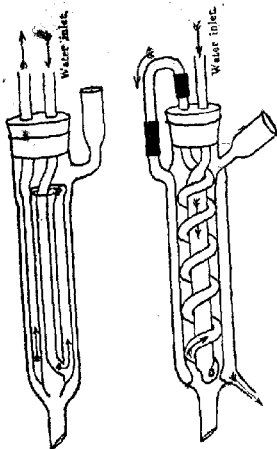
W. R.

An Apparatus for Extraction by Hot Ether. WILHELM KULKA (*Biochem. Zeitsch.*, 1908, 13, 134—137).—An extraction apparatus somewhat similar to Soxhlet's, but so modified that the vessel containing the substance to be extracted is surrounded by the ether vapour, its contents therefore being nearly at the temperature of boiling ether (about 30°).

S. B. S.

New Forms of Condenser. H. STOLTZENBERG (*Chem. Zentr.*, 1908, ii, 277; from *Zeitsch. Chem. Appar.*, 1908, 3, 243—245).—The condensers described have the advantage of exposing four cooling

surfaces to the vapour, are light and compact, and may be readily cleansed by removing the inner portion with the stopper. The accompanying figures should explain their construction. J. V. E.



A New Aspirating Reflux Condenser. HENRI VIGREUX (*Bull. Soc. chim.*, 1908, [iv], 3, 855—857).

—This consists of a Liebig condenser in which the water outflow pipe is fused to a small aspirator acting on the principle of an ordinary filter-pump. The air-exhaust tube of this aspirator is connected with the upper end of the inner condensing tube. The latter is provided on its inner surface with points and mounds in order to facilitate condensation. The effect of a current of water through the condenser envelope is thus to pro-

duce an upward current of air in the inner condensing tube, and it is claimed that such a condenser placed above an open vessel (basin, beaker or flask) containing the boiling liquid condenses the whole of the vapours, returning the condensed liquid to the vessel even when the liquid consists of ether, alcohol, light petroleum, or carbon disulphide. The apparatus is also of use in reactions involving hydrogen chloride or bromine or iodine vapours. E. H.

A New Aspirating Condenser and Recuperator for Rapid Evaporations. HENRI VIGREUX (*Bull. Soc. chim.*, 1908, [iv], 3, 858).—This is simply the condenser described previously (preceding abstract), of which the inner tube is provided, immediately below the outer jacket, with a side-tube arranged to collect the condensed liquid and to convey it through a trap (to prevent the aspirator from drawing air through this side-tube) into a receiver. Solutions in ether, light petroleum, benzene, or carbon disulphide can be thereby rapidly evaporated without risk of ignition. The lower end of the condenser should be placed slightly above the surface of the liquid when the latter is contained in a basin, and in the middle of the neck of a flask when this vessel is used. E. H.

A Simple Form of Sprengel Vacuum Pump. JAMES ARTHUR POLLOCK (*J. Roy. Soc., New South Wales*, 1907, 41, 140—143).—A modified short fall Sprengel pump, in which the raising of the mercury is effected by evaporating the mercury at a lower and condensing it at a

higher level; the mercury on reaching the bottom of the fall tube enters a horizontal tube, the farther end of which is bent vertically upwards; this vertical limb bears a bulb in which the mercury is boiled, the vapours being condensed by passing through a Liebig's condenser the lower end of which is connected to the fall tube. The apparatus must be used in connexion with an efficient auxiliary pump. P. H.

Turbine Funnel for Mixing Solutions. A. GAWALOWSKI (*Zeitsch. anal. Chem.*, 1908, 47, 697).—The apparatus is intended for the purpose of mixing a reagent with a solution. It consists of a funnel the stem of which is provided at the bottom with two arms placed at right-angles to the stem and ending in fine curved jets. A glass bulb is attached to the stem just below the funnel, and acts as a float, whilst the apparatus is kept in an upright position by means of a small quantity of mercury contained in a bulb below the two arms. The apparatus is floated in a solution contained in a beaker, and the reagent is poured into the funnel; as it escapes into the solution through the jets, the apparatus is caused to revolve, and the reagent and solution are thus mixed. W. P. S.

Inorganic Chemistry.

Catalysis of Hydrogen Peroxide by Iodine and Iodine Ions. EMIL ABEL (*Zeitsch. Elektrochem.*, 1908, 14, 598—607).—The reactions between hydrogen peroxide and iodine or iodine ions are: (1) $\text{H}_2\text{O}_2 + \text{I}_2 = 2\text{H}^+ + 2\text{I}^- + \text{O}_2$ and (2) $\text{H}_2\text{O}_2 + 2\text{I}^- + 2\text{H}^+ = 2\text{H}_2\text{O} + \text{I}_2$. The first is very rapid in alkaline solutions, and the second is rapid in acid solutions; in solutions containing a suitable small quantity of hydrogen ions, the two may take place with equal velocity, and the result is then simply decomposition of hydrogen peroxide, the concentrations of the iodine and iodine ions remaining unchanged. The ratio between the quantities of iodine and iodine ions which must be present in order to make the two reactions proceed equally fast depends on the concentration of the hydrogen ions. A large number of experiments, in which the concentration of the hydrogen ions is kept constant by adding acetic acid and sodium acetate to the solutions, are made which are in complete agreement with these views.

Bredig and Walton's observations (Abstr., 1904, ii, 319) on the catalysis of hydrogen peroxide by neutral iodides are explained by the same reactions, for in this case when reaction (2) has proceeded to a very small extent, the solution becomes alkaline and reaction (1) then proceeds exceedingly fast, the stage at which the two have equal velocity is therefore attained when a very small quantity of iodine has been liberated.

T. E.

Non-existence of a Polymeride of Chlorine. E. BRINER and E. DURAND (*Zeitsch. Elektrochem.*, 1908, 14, 796—797).—The authors have investigated the effect of the silent discharge on carefully-purified chlorine at a temperature but little above its boiling point. The experimental arrangements could have detected a contraction of 1/2000 of the volume of the gas, but no change was observed. The activity of chlorine observed by Kellner (*Abstr.*, 1902, ii, 649) and Russ (*Abstr.*, 1905, ii, 381) is possibly due to ozone or oxides of chlorine.

T. E.

Absolute Density of Hydrogen Sulphide. GEORGES BAUME and F. LOUIS PÉROT (*J. Chim. Phys.*, 1908, 6, 610—619).—Hydrogen sulphide from several sources, purified by fractional distillation, was employed. The density was determined by the method already described (this vol., ii, 372), except that an oil-gauge was interposed between the gas and the mercury.

The mean value of nearly twenty experiments for the weight of a litre of the gas at 0° and 760 mm. is 1.5392 grams. From this result, the atomic weight of sulphur has been calculated by Guye's method from the critical constants. The mean result is $S = 32.070$ ($H = 1.00775$), in excellent agreement with the gravimetric determinations of Richards and Jones (*Abstr.*, 1907, ii, 685).

G. S.

Preparation of Azoimide. JOHANNES THIELE (*Ber.*, 1908, 41, 2681—2683).—Azoimide is obtained in good yield by the action of ethyl nitrite on hydrazine in presence of alkali. Hydrazine hydrate (1 mol.) is mixed with 4*N* sodium methoxide ($1\frac{1}{2}$ mols.), ethyl nitrite ($1\frac{1}{2}$ mols.), and ether, and the mixture allowed to remain, first in ice, and then at the ordinary temperature. After twenty-four hours, the sodium azoimide is collected and washed. The yield is nearly quantitative. Instead of the hydrate, hydrazine sulphate may be used; this is ground with $3\frac{1}{2}$ mols. of sodium methoxide for six hours, the sodium sulphate filtered off, and the filtrate treated with ethyl nitrite and ether. For the direct preparation of free azoimide, hydrazine sulphate is shaken for six hours with aqueous sodium hydroxide and ethyl nitrite; in this way, 80—84% of the hydrazine is converted into azoimide. Sodium azoimide is also formed by heating hydrazine hydrate with aqueous sodium nitrite, preferably, in presence of hydrazine sulphate or sodium hydroxide, but the reaction is slow and incomplete.

J. C. C.

Formation of Nitric Oxide in the High Tension Arc. FRITZ HABER and ADOLF KOENIG (*Zeitsch. Elektrochem.*, 1908, 14, 689—695).—The authors have continued their experiments (this vol., ii, 34) by passing the high tension arc through nitric oxide at about 100 mm. pressure. A platinum wire of 0.01 mm. diameter, stretched across the quartz tube in which the arc is produced, melts when the current is increased to 0.2 ampere. The temperature is therefore not much above the melting point of platinum. Using the same current and passing the gas through the tube at the same rate, the percentage of nitric oxide (about 14.5) leaving the arc is the same whether nitric oxide is used

or a mixture of equal volumes of oxygen and nitrogen. The temperature is at most some hundreds of degrees above the melting point of platinum, and the equilibrium is therefore electrical, that is, due to ionic collisions, and not thermal.

T. E.

Interaction of Nitric Oxide and Oxygen. WILHELM HOLWECH (*Zeitsch. angew. Chem.*, 1908, 21, 2131—2135).—Mandl and Russ's observations (this vol., ii, 272) suggest that nitric oxide reacts only slowly with pure oxygen, the reaction being accelerated by the presence of catalysts. As this is of great importance for the practical working up of the nitric oxide which is formed in the electric furnace, the present author has endeavoured to prepare a non-reacting mixture of nitric oxide and ozonised oxygen with the object of studying the effect on it of catalysts. The apparatus employed is figured, and the results of the experiments represented by curves. It is found that 2 vols. of nitric oxide react with 1 vol. of oxygen almost completely at the atmospheric pressure with formation of NO_2 and N_2O_4 . The velocity of the reaction, by constant volume, is approximately the same for oxygen prepared from air by Linde's method, ozonised oxygen, oxygen from barium peroxide, and oxygen from potassium dichromate and sulphuric acid.

G. Y.

Action of Nitrous Gases and Oxygen on Water. FRITZ FORSTER and M. KOCH (*Zeitsch. angew. Chem.*, 1908, 21, 2161—2172).—A résumé is given of the work of various authors on the preparation of nitric and nitrous acids from the nitric oxide formed by combustion of atmospheric nitrogen. It is pointed out that the concentration of the nitric acid formed by the action of a mixture of nitrogen dioxide and oxygen on water must reach a limit, which is a function of the velocity with which the equilibrium $2\text{NO} + \text{H}_2\text{O} \rightleftharpoons \text{HNO}_2 + \text{HNO}_3$ is reached, and, therefore, of the concentration of the nitrogen dioxide, since the lower the partial pressure of the dioxide the smaller will be the concentration of its solution in the water, and hence the velocity of its reaction with the water. On the other hand, the larger volume of undissolved gases passing through the solution will cause more rapid evaporation, in which the nitric acid takes part even at concentrations far below 68%. The maximum concentration of nitric acid which can be obtained by the action of a mixture of nitrogen dioxide and oxygen on water, must lie about this percentage, since, when solutions containing higher percentages are evaporated, the nitric acid volatilises more rapidly than the water. For the same reasons, the limit concentration must be the lower the more rapid the current of gas. These views are confirmed by the results now obtained on studying the action on water of mixtures of nitrogen dioxide with oxygen and with air in varying proportions. The apparatus employed is figured, and the results are tabulated.

The formation of nitric acid takes place fairly rapidly until the solution contains about 54% of acid, a concentration corresponding with the composition $\text{HNO}_3 \cdot 3\text{H}_2\text{O}$, when, in consequence of the greatly diminished amount of free water, the reaction becomes greatly retarded. It is found that, whilst the concentration of a 64.22% acid is raised to 67.6% by the action at 0° of a mixture containing 63.2% by volume of

nitrogen dioxide, at 60° the concentration is raised by the same mixture, passing into the nitric acid at the same rate and for the same period, only to 65.2%. The higher temperature is the more unfavourable to the reaction, which is, therefore, probably one between water, oxygen, and the bimolecular dioxide, N_2O_4 .

The influence of the partial pressure is shown in that, whilst almost the whole of the dioxide of a mixture of 2.5 vols. of nitrogen dioxide with 6.0 vols. of oxygen is absorbed by water with formation of a 37% acid, under the same conditions only 53% of the dioxide of a mixture of 2.5 vols. of dioxide and 122 vols. of oxygen is absorbed with formation of a 27.73% acid.

Whilst at low concentrations the velocity of the reaction is affected only slightly by the rate at which the gases are passed into the water, a marked effect is produced at concentrations over 50% of acid. Equal volumes of a mixture containing 5.0 vols. of dioxide and about 12 vols. of oxygen, when passed into water at the rates of 4.25, 8.5, and 17.0 litres per hour, yielded 58.78%, 55.36%, and 53.95% acids respectively. The bearing of the experimental results on the behaviour of nitrous acid also is discussed.

G. Y.

Molecular Weight and Hydrates of Hypophosphoric Acid.

ARTHUR ROSENHEIM and MAX PRITZE (*Ber.*, 1908, 41, 2708—2711).

—The controversy between Rosenheim, Stadler, and Jacobsohn (*Abstr.*, 1906, ii, 744) and Parravano and Marini (*Abstr.*, 1906, ii, 744) as to the molecular weight of hypophosphoric acid has led the authors to determine the molecular weights of the methyl and ethyl esters of pyro- and of hypo-phosphoric acids in methyl or ethyl iodide by the ebullioscopic method, with results which indicate that the formulae of the acids are $H_4P_2O_7$ and $H_4P_3O_{10}$ respectively.

The monohydrate, $H_2PO_6 \cdot H_2O$, m. p. 62°, appears to be the only hydrate stable between 0° and 60°; Joly's anhydride (*Abstr.*, 1886, 662), or Sönger's hydrate, $H_2PO_{3.2} \cdot \frac{1}{2}H_2O$, could not be obtained.

C. S.

Coagulation of [Colloidal] Arsenious Sulphide by Barium Chloride. JACQUES DUCLAUX (*J. Chim. Phys.*, 1908, 6, 592—595).—

When a colloidal solution of arsenious sulphide is precipitated by barium chloride, part of the barium is carried down with the precipitate and the remaining solution is acid. This is usually accounted for on physical lines as being due to absorption of the barium by the sulphide with consequent hydrolysis of the salt (Whitney and Ober, *Abstr.*, 1902, ii, 65), although Linder and Pictou (*Trans.*, 1895, 67, 63) showed that the sulphide usually contains excess of hydrogen sulphide, and that in the presence of barium chloride barium sulphide is formed and hydrogen chloride set free.

The author now shows that when a colloidal solution of arsenious sulphide containing excess of hydrogen sulphide, which has been kept well stoppered for five years, is precipitated with barium chloride and filtered, the filtrate contains no hydrogen sulphide, but a certain amount of arsenious acid; the latter corresponds approximately with the amount of hydrogen chloride in the filtrate, and therefore with that of the barium absorbed. The author considers that neither of the above

explanations of the phenomenon are satisfactory; it is probable that there is double decomposition between the barium chloride and the sulphide, perhaps by replacement of an AsO group existing in the sulphide by barium. G. S.

Preparation of Carbon Monoxide. ERWIN RUFF (*Chem. Zeit.*, 1908, 32, 983).—A known weight of sulphuric acid is heated in a generating flask to 100°, and an equal weight of 98% formic acid is dropped in from a stoppered funnel. A regular stream of pure carbon monoxide is thus evolved; when the action slackens, a gentle heat is again applied by means of a small flame. L. DE K.

Simple Arrangement for Filling Glass Tubes with Liquid Carbon Dioxide. ALFRED THIEL (*Chem. Zentr.*, 1908, ii, 470—471; from *Zeitsch. Chem. Appar.*, 1908, 3, 240—243).—An arrangement is described for filling evacuated glass tubes with carbon dioxide to allow of the demonstration of the critical temperature of this gas. The glass tube to be filled is placed in a cooling bath of solid carbon dioxide and ether, and is connected by a stout capillary tube to a compression tube, which is regulated by an adjustable head of mercury. Air-free carbon dioxide is admitted to the compression tube by lowering the head of mercury, when, by closing a tap on the inlet tube and opening a tap connecting with the exhausted condensing tube and increasing the head of mercury, the enclosed carbon dioxide is transferred to the cooled tube, where it is condensed. By repeating this operation several times, any required quantity of carbon dioxide may be condensed in an expeditious manner. J. V. E.

Argon in Radioactive Zirconium Minerals. A. VON ANTROPOFF (*Zeitsch. Elektrochem.*, 1908, 14, 585—586).—The gas extracted from a feebly radioactive Brazilian mineral containing 97% of zirconia consisted mainly of carbon dioxide with smaller quantities of hydrogen and nitrogen and traces of helium and argon. This is the second mineral in which argon has been found; the first was also a radioactive zirconium compound (*Trans.*, 1906, 89, 1568). T. E.

Analysis of the Atmospheric Gases Non-liquefiable in Liquid Air. FRÉD BORDAS and TOUPLAIN (*Compt. rend.*, 1908, 147, 591—594).—A modified form of the apparatus described previously (this vol., ii, 430) has been employed for the analysis of the gases which escape liquefaction when liquid air is distilled and the oxygen and nitrogen condensed. A table is given of the wave-lengths of the lines observed in the spectrum of this gas after removal of all the oxygen and nitrogen. This includes, besides those observed by Baly for neon and helium, the red lines of wave-lengths 7267, 7050, 6943, 6732, 6610, 6572, 6535, 6509; the orange lines, 5905, 5890; the yellow lines, 5820, 5808, 5765, and the green lines, 5750, 5722, 5694, 5659, 5569, 5441, 5407, 5348, 5336, which the authors attribute to neon, but which have not been observed previously.

The conclusion is drawn that the gases in the atmosphere non-liquefiable in liquid air consist of neon and helium. E. H.

Liquefaction of Helium. H. KAMERLINGH ONNES (*Proc. K. Akad. Wetensch. Amsterdam*, 1908, 11, 168—185; *Compt. rend.*, 1908, 147, 421—424).—The actual work carried out with the object of liquefying helium was preceded by a study of the isothermal lines for such temperatures as can be attained by the use of liquid hydrogen. The results of this study made it very probable that the Joule-Kelvin effect would not only give a decided cooling at the melting point of hydrogen, but that this would be sufficient to make a Linde-Hampson process successful. The construction of the helium liquefier was therefore an imitation of the hydrogen liquefier described in a previous communication.

The helium employed was obtained by heating monazite sand. The gas was then successively exploded with oxygen, cooled with liquid air, compressed, and led over charcoal at the temperature of liquid air. It was then passed through a column of hot copper oxide, compressed over charcoal at the temperature of liquid air, and further led over charcoal under pressure at the temperature of liquid hydrogen, until the gas which had been absorbed in the charcoal and then separately collected no longer contained any appreciable impurity.

On the day before the final experiment, 75 litres of liquid air were prepared, and on the day itself, operations began with the preparation of 20 litres of liquid hydrogen. When the helium (200 litres) had been circulating in the liquefier for about three hours, more than 60 c.c. of liquid helium was obtained. In consequence of the extreme purity of the helium, and by virtue of the special contrivances adopted, the apparatus remained perfectly transparent during the whole experiment.

The liquid helium looks as if it was almost at its critical temperature, and its surface stands out like the edge of a knife against the glass. Evaporation under diminished pressure did not lead to solidification, and the triple point pressure is certainly below 1 cm. The temperature reached in this evaporation was probably about 3° absolute.

The boiling point was found to be 4.3° absolute, but a slight correction is suggested which would make it about 4.5°. The density of the liquid is 0.15; the ratio of the density of the vapour to that of the liquid is 1:11 at the boiling point, indicating that the critical temperature is not much higher than 5° absolute, and that the critical pressure is not much more than 2.3 atmospheres.

The value of a , the constant of van der Waals' equation, appears for helium to be about 0.00005, and the value of b is probably about double the value which was expected (0.0005). J. C. P.

Preparation of Colourless Alcoholic Potassium Hydroxide ADOLF HALLA (*Chem. Zeit.*, 1908, 32, 890).—Thirty grams of pure stick potassium hydroxide are placed in a bottle with one litre of 95° alcohol, and left at atmospheric temperature until solution is effected the bottle being shaken from time to time. The clear supernatant liquid is then carefully siphoned off into another bottle, which is stoppered and exposed to diffused daylight. The solution is thereby

bleached in the course of a few days, and remains colourless so long as it is kept in diffused daylight. P. H.

The Partition of Silver between Zinc and Lead. G. N. POTDAR (*J. Coll. Sci. Tokyo*, 1908, 25, ix, 1—13. Compare Wright and Thompson, *Abstr.*, 1890, 336).—Silver-lead alloys were melted with zinc in an atmosphere of carbon dioxide, the temperature being maintained at 540° by means of a bath of boiling phosphoric sulphide. After stirring, the mass was allowed to remain fluid for five and a-half hours, rapidly cooled, and the silver in the two solidified layers estimated. The whole of the zinc layer has to be taken for analysis, as the distribution of zinc in it is far from uniform, owing perhaps to segregation during cooling. At 540° , silver is about 300 times more soluble in zinc than in lead, and this partition-coefficient is practically independent of the concentration of the silver.

In Parkes' process for the desilverisation of lead, the solid zinc scum is removed at $325\text{--}360^{\circ}$. Experiments at this temperature indicate that the removal of silver by the zinc is practically complete, the partition-coefficient at 358° varying in three trials from 1300 to 5700. C. H. D.

The Silver Hydrogel in Photographic Films. LÜPPO-CHAMER (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 135—136).—The peculiarities of the silver deposits obtained with different developers, and with gelatin or collodion films, are explained by the more or less completely colloidal nature of the silver, and by adsorption of different substances from the developers (compare this vol., ii, 841). T. E.

Use of Metallic Deposits in Metallography. II. FEDERICO GIOLITTI (*Gazzetta*, 1908, 38, ii, 352—357).—The author has applied the method devised by him (*Abstr.*, 1906, ii, 759) to the qualitative study of alloys constituted of solid solutions. When a polished surface of a binary alloy containing mixed crystals either of two metals or of one of them and a compound of the two is immersed in a solution containing ions of the more electronegative or more noble of the two metals, differences of potential are established between the various portions of the surface. The cations of the solution will be deposited in the metallic state on the different parts of the section with a velocity increasing with the concentration of the more electronegative metal. By varying the concentration of the solution and the duration of immersion, or by observing the section under the microscope, the gradual and continuous variations of the metallic deposit on that part of the surface occupied by the mixed crystals can be followed. These variations, which correspond with differences in the concentration of the more noble metal in the mixed crystals, give an immediate indication that the constituent in question is a solid solution, and also permit the observer to judge the heterogeneity of concentration of this solid solution. Various bronzes have been studied in this way, microphotographs of the different sections after treatment with chromic oxide or with copper sulphate solution being given. T. H. P.

Magnesium Oxychloride Formed by Electrolysis of the Residual Solutions from the Manufacture of Potassium Chloride, and its Importance for the Preparation of Bromine by Kossuth and Mehns's Method. HANS HOF (*Chem. Zeit.*, 1908, 32, 993).—It has been found that the yield of free bromine, obtained by electrolysis of the residual solutions from the manufacture of potassium chloride, increases with the concentration of the solution electrolysed. On dilution with water, the electrolysed solution containing free bromine becomes gradually less yellow, and finally colourless, when the solution is found to contain only combined bromine; this may be again liberated by sulphuric acid. If the bromine is first removed by means of an organic solvent, the electrolysed solution, on dilution, becomes alkaline and opaque, in consequence of the separation of magnesium hydroxide. It is considered that magnesium hydroxide, formed during the electrolysis, remains dissolved in the concentrated magnesium chloride solution, forming a basic salt. This is decomposed by water with liberation of magnesium hydroxide, which then reacts with the free bromine. In agreement with this, analytical figures are given, showing that the proportion of bromine present as bromate to the free bromine increases with the dilution of the electrolysed solution.

By electrolysis of a concentrated solution of magnesium chloride, the author has obtained a neutral solution which does not react with bromine. When diluted, it becomes alkaline, and deposits magnesium hydroxide. The concentrated solution slowly, deposits a white, amorphous mass, $\text{MgCl}_2 \cdot 5\text{MgO}$ (Bender, *Annalen*, 1871, 159, 341; Davis, *Chem News*, 1872, 25, 258), which is stable at 160° and yields magnesium chloride when treated with cold water, but is completely decomposed only by boiling water. G. Y.

Electrolytic Oxidation of Copper. F. SCHMIEDT (*Chem. Zentr.*, 1908, ii, 386; from *Elektrochem. Zeitsch.*, 1908, 15, 53—56).—The author has repeated the work of Müller and Spitzer (*Abstr.*, 1907, ii, 174), and finds that, besides cupric oxide and copper peroxide, there is also formed cuprous oxide, which, however, becomes decomposed by the hydrogen peroxide that is also produced. Copper peroxide is formed in largest quantities at the beginning of the electrolysis, when the temperature is low. J. V. E.

Alloys of Copper and Tin. FEDERICO GIOLITTI and G. TAVANTI (*Gazzetta*, 1908, 38, ii, 209—239).—The results obtained by the authors in studying the freezing points and microscopic appearance of the copper-tin alloys agree, in general, with those of Heycock and Neville (*Abstr.*, 1901, ii, 508; 1902, ii, 261). The freezing-point diagram of these alloys is divided clearly into two parts, the first comprising alloys containing from 0 to 38.34% of tin, and the second, those containing more than 38.34% of tin. Thus the diagram is a complex one, resulting from the combination of the equilibrium diagrams of $\text{Cu-Cu}_3\text{Sn}$ and $\text{Cu}_3\text{Sn-Sn}$. The diagram is discussed in detail. T. H. P.

Copper and Iron Salts in Presence of Alkalis and Acids. HERMANN FRISCHER (*Chem. Zeit.*, 1908, 32, 1005—1006).—Experiments are described to show that the action of sodium hydroxide on a mixture of ferrous and cupric sulphates takes place, contrary to Millberg's supposition (*Chem. Zeit.*, 1906, 30, 511; 1907, 31, 1143), according to the following equations: (1) $2\text{CuSO}_4 + 4\text{NaHO} = 2\text{Cu}(\text{OH})_2 + 2\text{Na}_2\text{SO}_4$, and (2) $2\text{FeSO}_4 + 2\text{Cu}(\text{OH})_2 = \text{Fe}_2(\text{OH})_4\text{SO}_4 + \text{Cu}_2\text{SO}$. The action of air on the resulting mixture leads to the formation of ferric hydroxide and cupric sulphate, the whole reaction being represented by the equation: $2\text{FeSO}_4 + 2\text{CuSO}_4 + 4\text{NaHO} + \text{H}_2\text{O} + \text{O} = 2\text{Fe}(\text{OH})_3 + 2\text{CuSO}_4 + 2\text{Na}_2\text{SO}_4$. Any further addition of alkali must result in precipitation of copper hydroxide. As the equation shows, the copper sulphate acts as the oxygen carrier; in agreement with this view, if potassium sodium tartrate and an excess of alkali are added to the mixture of ferrous and cupric salts, cuprous hydroxide is precipitated in the cold, and on heating is converted into the red anhydride, whilst the ferric salt remains in solution. If a sufficient excess of ferrous salt is present, the cuprous hydroxide is reduced to metallic copper. This reaction may be employed for the detection of ferrous salts.

If a ferrous salt is added to an ammoniacal solution of a cupric salt, ferric hydroxide is precipitated, whilst the blue colour of the solution gradually disappears; the cuprous salt remaining in solution is rapidly oxidised by air. In this case, also, the precipitation of the ferric hydroxide may be prevented by addition of a tartrate or a citrate.

The behaviour of copper and iron salts in presence of ammonia affords a ready means of preparing cuprous solutions for the absorption of carbon monoxide in gas analysis. It is evident, moreover, that, in the separation of iron and copper by means of ammonia, a preliminary oxidation of ferrous salts is unnecessary if the amount of copper present is equal to, or greater than, that of the iron. On the other hand, if the copper is to be estimated colorimetrically, the ferrous salts present must first be oxidised.

In acid solution, cuprous salts are oxidised at the expense of ferric salts, according to the equation: $\text{Cu}_2\text{O} + \text{Fe}_2\text{O}_3 = 2\text{CuO} + 2\text{FeO}$.

G. Y.

Equilibrium in the Reaction between Lead Sulphide and Its Oxidation Products. III. RUDOLF SCHENCK and W. RASSBRACH (*Ber.*, 1908, 41, 2917—2925).—Previous investigations (Abstr., 1907, ii, 546, 619) have shown that the metallurgically important reactions: (I) $\text{PbS} + \text{PbSO}_4 = 2\text{Pb} + 2\text{SO}_2$; (II) $\text{PbS} + 2\text{PbO} = 3\text{Pb} + \text{SO}_2$, are reversible, the equilibrium being conditioned by a definite pressure of the sulphur dioxide, which is dependent on the temperature only. In addition to the two temperature-pressure curves so obtained, a third was indicated, the cause of which forms the main object of the present investigation. The result shows that a basic sulphate, $\text{PbSO}_4 \cdot \text{PbO}$, is formed, which, setting up its own reversible reaction, $2[\text{PbSO}_4 \cdot \text{PbO}] + \text{PbS} \rightleftharpoons 7\text{Pb} + 5\text{SO}_2$, accounts for the existence of the third temperature-pressure curve. These three curves divide the whole temperature-pressure diagram into four fields. The final product of all reactions in the first field is lead sulphate, in the second the basic sulphate, and in

the third lead oxide; these three stable phases do not evolve sulphur dioxide with lead sulphide. In the fourth field, no compound containing oxygen is stable with lead sulphide, an impure metal being obtained with evolution of sulphur dioxide. These conclusions are deduced from observations at temperatures below 820° ; above this temperature, complications are introduced, owing to the mutual solubility of lead oxide and lead sulphate.

The existence of the basic sulphate, $\text{PbSO}_4 \cdot \text{PbO}$, is proved as follows. Mixtures of lead sulphate and lead oxide in known proportions are heated to 1020° , and allowed to cool. The freezing-point curve, consisting of many branches, indicates the existence of many compounds of lead sulphate and lead oxide which do not form mixed crystals with one another or with either of the components. Distinct maxima are observed at two points: (966° , 40% PbO) and (951° , 61.5% PbO); the compositions of the mixture at these points correspond with the formulae $\text{PbSO}_4 \cdot \text{PbO}$ and $\text{PbSO}_4 \cdot 2\text{PbO}$ respectively. The existence of a third basic sulphate, $\text{PbSO}_4 \cdot 3\text{PbO}$, is indicated by the curve, which, however, is only stable below 880° ; above this temperature, it passes into $\text{PbSO}_4 \cdot 2\text{PbO}$. The presence of these basic sulphates in the fused masses containing 40–70% lead oxide is indicated by the appearance of long, transparent needles. The only basic sulphate giving in the presence of lead sulphide and a little lead a well-defined temperature-pressure curve is $\text{PbSO}_4 \cdot \text{PbO}$, as already mentioned; the others react too slowly for any trustworthy data to be obtained.

The following facts have been observed during the investigation. Lead sulphate does not melt below 1100° (compare Ramsay and Eumorfopoulos, *Phil. Mag.*, 1896, [v], 41, 360); an exact determination cannot be made, owing to dissociation into sulphur trioxide. Pure lead sulphate shows a transition point at 850° . Mixtures of lead oxide and sulphate, rich in the former, change in volume at the ordinary temperature, the original dense mass swelling up and crumbling to a coarse powder, in which scales of lead oxide can be detected. The force exerted during the expansion distorted the containing platinum crucible. C. S.

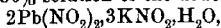
Compounds of Lead with Nitrous Acid. ALBERTO CHILLESOTTI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 173–183, 288–295. Compare this vol., ii, 845).—By measuring the *E.M.F.* of cells consisting of two lead electrodes, one immersed in a solution of a lead salt containing a known concentration of lead ions, and the other in a 0.1*N* lead nitrite solution, it is found that the concentration of the lead ions in the latter is very nearly equal to their concentration in a lead chloride solution of corresponding concentration (compare Abegg and Lahendzinski, *Abstr.*, 1904, ii, 241). The lower conductivity of lead nitrite solutions, compared with those of lead chloride, is hence due, not to a less degree of dissociation (*loc. cit.*), but to a difference in the mobility of the complex ions.

The conductivity of lead nitrite solutions undergoes a gradual increase, owing to decomposition of the nitrite according to the equation: $3\text{Pb}(\text{NO}_2)_2 + 2\text{H}_2\text{O} = \text{Pb}(\text{NO}_3)_2 + 2\text{Pb}(\text{OH})_2 + 4\text{NO}$; probably a small amount of secondary decomposition occurs with evolution of

nitrous oxide. The solutions examined, after boiling and cooling, deposit nacreous scales of $\text{Pb}(\text{NO}_2)_2 \cdot \text{Pb}(\text{OH})_2 \cdot \text{H}_2\text{O}$ or $\text{Pb}(\text{NO}_2)_2 \cdot \text{Pb}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

Attempts to prepare a double nitrite of lead and potassium by mixing hot concentrated solutions of lead acetate and potassium nitrite in various proportions led, in all cases, to crystalline compounds, in which the ratio $\text{Pb} : \text{NO}_2$ lies between 1:3 and 1:4; the excess of lead oxide present is due probably to decomposition of the lead nitrite.

By the gradual addition of freshly precipitated and washed lead hydroxide to a boiling 30% solution of the double nitrite,



the author has prepared the basic lead nitrite, $\text{Pb}(\text{NO}_2)_2 \cdot \text{Pb}(\text{OH})_2 \cdot \text{H}_2\text{O}$, which can be obtained in the pure, non-hydrolysed condition by gently warming the impure product with a cold saturated, crude solution of the salt, and allowing to crystallise. Under certain conditions, this basic salt separates in the form $\text{Pb}(\text{NO}_2)_2 \cdot \text{Pb}(\text{OH})_2$. Study of the action of lead on a lead nitrate solution leads to the confirmation of the existence of the compound $4\text{PbO} \cdot \text{N}_2\text{O}_5 \cdot \text{H}_2\text{O}$, prepared by Chevreul (*Ann. Chim.*, 1812, 83, 72) and others.

T. H. P.

Coagulation of Colloidal Aluminium Hydroxide by Electrolytes. SHIN-ICHI KAWAMURA (*J. Coll. Sci. Tôkyô*, 1908, 25, viii, 1—29).—It has been found possible to determine the degree of coagulation of a colloidal solution of aluminium hydroxide, produced by various electrolytes, by measuring the viscosities of the solutions. The general relations found are not affected to any marked degree by the temperature, concentration of the colloidal solution, or duration of the experiment, and may be summarised as follows: (1) In those cases where coagulation occurs, the relative viscosity shows a marked rise only when the concentration of the electrolytes reaches certain values. From this point, the viscosity increases with the increasing concentration of the electrolyte to a certain limit, and then ceases to be influenced by the further addition of the electrolyte. In some cases, the degree of coagulation increases suddenly, and in others, more gradually, with the increasing concentration of the electrolyte. (2) The colloidal solution of aluminium hydroxide shows anodic cathaphoresis; consequently, in accordance with Hardy's rule (compare Abstr., 1899, ii, 567; 1903, ii, 469; 1906, i, 121), only anions are found to exercise coagulating power. (3) With but few exceptions, the coagulating power of an electrolyte increases rapidly with the increasing valency of the anion; this is in agreement with Schulze's rule (compare Abstr., 1883, 295). (4) The highest degree of coagulation produced by various electrolytes differs considerably in different cases, and appears to be a characteristic of the electrolyte.

W. H. G.

Crystallised Iron from a Foundry at Teschen, Austria. FELIX CORNU (*Centr. Min.*, 1908, 545—546).—The crystals are about 3 mm. across, and have the form of distorted octahedra grouped along

the three cubic axes (similar to the well-known skeletal growths of magnetite). Anal. I of the crystals, and II of the ground mass of iron in which they are embedded.

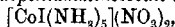
	Fe.	Si.	P.	S.	Cu.	Mn.	C.	O.
I.	95.32	0.22	0.16	—	—	1.24	1.80	1.26
II.	95.91	0.19	0.24	0.30	0.10	1.46	1.80	—

L. J. S.

Nature of the Pseudo-solutions of Ferric Hydroxide. III. FEDERICO GIOLITTI (*Gazzetta*, 1908, 38, ii, 252—258. Compare Abstr., 1906, ii, 857).—The typical or stable ferric hydrosol, prepared by treating washed precipitated ferric hydroxide with acetic acid after keeping it in contact with water for several months and dissolving in water the residue insoluble in acetic acid, is either a ferric hydroxide containing less water than the other hydrosols and the ordinary gelatinous hydroxide, or possibly anhydrous ferric oxide; the latter view is supported by the comparative difficulty with which this hydrosol is dissolved by acids. The hydrosol of Graham, prepared by dialysis, also changes gradually into the stable hydrosol.

T. H. P.

Iodopentamminecobalt Salts. ALFRED WERNER (*Ber.*, 1908, 41, 3007—3015).—The author finds that the substances obtained by Sand and Böckmann (this vol., ii, 44) by the action of iodine on the black nitrosopentamminecobalt salts, to which they ascribe the constitution of a complex metal ammonia containing cobalt atoms with co-ordination numbers 7 and 8, are, in reality, members of the hitherto unknown series of iodopentamminecobalt salts. For example, the product of the action of iodine on black nitrosopentamminecobalt nitrate is a mixture of *iodopentamminecobalt nitrate*,



with a little xantho-nitrate; the separation of the two is effected through the dichromates. By treatment with silver nitrate, the former yields aquopentamminecobalt nitrate, thus proving its constitution.

Members of the new series are readily obtained by heating aquopentamminecobalt iodide at 80°. Water is evolved, and the brown product, triturated with ammonium nitrate, yields iodopentamminecobalt nitrate. The *chloride*, *bromide*, *iodide*, and *dichromate* are described; they all have a green colour.

C. S.

Relationships between Nickel and Hydrogen. M. MAYER and V. ALTMAYER (*Ber.*, 1908, 41, 3062—3074).—The absorption of hydrogen by reduced nickel has been studied at temperatures between 360° and 560°, and at pressures between 1/15 and 4/5 of an atmosphere. The metal was obtained by the same method as was used by Sieverts (Abstr., 1907, ii, 741), but the conditions of absorption were different, as only small amounts (2.7 grams) of metal were used, and each experiment was allowed to proceed until equilibrium was attained. The amount of gas absorbed was much greater than in Sievert's experiments; thus at pressures between 1/15 and 4/5 of an

atmosphere, and a temperature of 360° , 1 vol. of the metal absorbs 5.5–50 volumes of gas. The isotherms representing the relationship between the pressure of the hydrogen and the relative number of atoms of hydrogen absorbed, are straight lines; thus indicating that Henry's law holds good, and that the system nickel-hydrogen is bivalent.

The rapidity with which equilibrium is established varies considerably with the amount of hydrogen present.

The solubility coefficient, that is, the ratio weight of absorbed hydrogen in 1 litre of nickel/weight of hydrogen in 1 litre of the gas volume, varies with the temperature.

	360° .	420° .	480° .	560° .
Ratio.....	297	302	306.4	299.1

J. J. S.

Decomposition of Chromic Acid by Hydrogen Peroxide

ERNST H. RIESENFELD and ALFRED WESCH (*Ber.*, 1908, 41, 2826–2835. Compare Abstr., 1905, ii, 825).—The decomposition of chromic acid by hydrogen peroxide was measured in presence of a varying excess of chromic acid. In spite of an excess of from one to one hundred times the theoretical, the amount of oxygen evolved remained constant, averaging 1.94 atoms per molecule of hydrogen peroxide. Decomposition of chromic acid in presence of excess of hydrogen peroxide and increasing amounts of sulphuric acid showed that up to a certain point the reaction remained unchanged, 1 atom of chromium corresponding with about 4.7 atoms of oxygen. Above 2.5*N* acid, a strength known to react with hydrogen peroxide to form Caro's acid, more oxygen than 5 atoms is evolved, due to a decomposition of the Caro's acid in presence of chromic salts. When the same change is studied in presence of excess of chromic acid, it is found to be far less dependent on the amount of sulphuric acid. Above 2.5*N*, Caro's acid is formed as before, with the result that slightly less hydrogen peroxide is available, and accordingly less oxygen than 2 atoms is liberated. The concentration of the sulphuric acid when chromic acid solution is dropped into hydrogen peroxide is thus without influence, and perchromic acid, H_2CrO_5 , is formed; likewise, when hydrogen peroxide is dropped into excess of a mixture of potassium dichromate and sulphuric acid, the excess of chromic acid is without influence.

Spitalsky's views (Abstr., 1907, ii, 338, 942) are adversely criticised.

E. F. A.

Preparation of Uranium. FEDERICO GIOLITTI and G. TAVANTI (*Gazzetta*, 1908, 38, ii, 239–251).—The authors have investigated the various methods of preparing uranium.

The "pure uranyl acetate" of Kahlbaum or Merck is, in reality, a double sodium uranyl acetate, $\text{UO}_2(\text{OAc})_2 \cdot \text{NaOAc} \cdot 2\text{H}_2\text{O}$, which, on ignition, yields sodium pyrouanate, $\text{Na}_2\text{U}_2\text{O}_7$, as sole product; the latter is also obtained by igniting other uranyl salts of volatile acids containing salts of the alkali metals. When sodium pyrouanate is

heated to redness in a current of hydrogen, it is reduced according to the following equation: $\text{Na}_2\text{U}_2\text{O}_7 + 2\text{H}_2 = 2\text{NaOH} + 2\text{UO}_2 + \text{H}_2\text{O}$, but it is found to be impossible to remove the sodium hydroxide by washing with water.

The oxide, U_3O_8 , can be obtained pure as follows: A solution of sodium uranyl acetate (100 grams) in a mixture of water (4 litres) and hydrochloric acid (50 c.c.) is precipitated by means of concentrated ammonia solution (300 c.c.), the precipitate being washed ten times by decantation with 8–10 litres of 2% ammonium chloride solution. The precipitate is then re-dissolved in hydrochloric acid, precipitated by means of ammonia solution, and washed as before, these operations being afterwards repeated a third time. On calcining the final precipitate, pure U_3O_8 , free from alkali, is obtained.

When a mixture of this oxide with excess of powdered aluminium is thrown on to the surface of fused aluminium maintained at a vivid red heat, not alloys of aluminium and uranium, mixed with alumina as stated by Moissan (Abstr., 1906, ii, 601), but a mixture of uranous oxide, alumina, and aluminium are obtained. When, however, a mixture of ferric oxide with 10% of the oxide, U_3O_8 , is intimately mixed with more than sufficient aluminium filings to reduce both the oxides, and the reaction started by means of barium peroxide and aluminium, the oxide of uranium is reduced to metallic uranium.

T. H. P.

New Element in Thorianite. MASATAKA OGAWA (*J. Coll. Sci. Tôkyô*, 1908, 25, xv, 1–11).—A new element, which it is proposed to name *nipponium* (Np), has been isolated from thorianite, reinite, and molybdenite. Its equivalent weight is about 50 and atomic weight about 100, so that this element probably fills the gap between molybdenum and ruthenium. Two *oxides* of the element appear to exist; the higher oxide is an acidic oxide, and is reduced by zinc and hydrochloric acid to the basic oxide; the latter oxide, which in the ordinary course of analysis accompanies aluminium oxide, is separated from this by conversion into a difficultly volatile chloride by heating in a current of chlorine and carbon tetrachloride vapour. The anhydrous *chloride* so formed dissolves in water to a pale green solution, and gives a characteristic line, having a wave-length of 4882, together with two other feebler lines, in the greenish-blue part of the spectrum. The *hydroxide*, precipitated by ammonia in the presence of ammonium chloride, is almost white with a pale yellow tinge, and dissolves in alkalis; it becomes nearly black when dried at 100°. The ignited oxide is brown, and is insoluble in acids. The yellowish-green solution of the hydroxide in hydrochloric acid gives a brown precipitate when boiled with sodium thiosulphate, and a greenish-black precipitate with ammonium sulphide. A solution of the chloride acidified with acetic acid gives a yellowish-brown precipitate with potassium chromate. A brown precipitate is obtained by passing carbon dioxide into a solution of the green mass formed by fusing the oxide with sodium carbonate and potassium nitrate; it dissolves readily in dilute hydrochloric acid. The aqueous solution of the fused mass does not yield a precipitate with ammonia or ammonium

sulphide, but the addition of acid to the solution containing ammonium sulphide produces a brown precipitate. W. H. G.

New Element Allied to Molybdenum. MASATAKA OGAWA (*J. Coll. Sci. Tokyo*, 1908, 25, xvi, 1—13).—Both molybdenite and thorianite contain a new element, closely allied to molybdenum, which is probably identical with the new tin-group element recently described by Miss Evans (*Trans.*, 1908, 93, 666). The equivalent of the element is about 16.7. It appears to form two oxides; the higher oxide resembles molybdenum trioxide, and yields *barium*, *lead*, and *silver* salts similar to the corresponding molybdates; the *mercurous* salt, however, forms golden-yellow crystals; the hydrated lower oxide (I) has a graphitic appearance, and is insoluble in hot dilute hydrochloric acid. The oxides are reduced by hydrogen to the *metal*, which is non-fusible at a red heat; it burns brilliantly in the air, forming the higher oxide. The new element differs from molybdenum in that it is soluble in hot concentrated hydrochloric acid, and gives no colour reaction when its higher oxide is reduced with zinc and hydrochloric acid, either in the presence or absence of potassium thiocyanate.

Thorianite appears to contain, in addition to this new element and nipponium (compare preceding abstract), yet another new element, the oxide of which is radioactive, and dissolves in nitric acid to a bluish-green solution. W. H. G.

A New Form of "Tin Plague." RUDOLF VON HASSLINGER (*Monatsh.*, 1908, 29, 787—790. Compare Cohen, *Abstr.*, 1900, ii, 212; this vol., ii, 858).—A tinned iron vessel, which had been for two years at 16—45°, showed a crystalline, brittle surface. Other masses of tin, inoculated with small portions of the crystalline tin, underwent the same change, the area affected increasing in diameter 3 to 5 mm. daily. No difference between experiments at 7°, 19°, and 37° could be noted. When tin-foil was inoculated, the change extended through the thickness of the foil. The crystalline tin preserves its appearance up to near the melting point, but becomes normal after melting and again solidifying. C. H. D.

Reduction of Vanadic Acid by Potassium Iodide. T. WARYSKI and B. MDIVANI (*Chem. Zentr.*, 1908, ii, 763; from *Mon. Sci.*, 1908, 22, [ii], 527—528).—With the object of ascertaining whether the reducing power of potassium iodide in an acid medium is dependent on the electrolytic dissociation of the acid employed, comparable tests have been made, using acetic acid and mono-, di-, and tri-chloroacetic acids. It is found that reduction is effected most readily in the presence of trichloroacetic acid, and least readily in presence of acetic acid. With trichloroacetic acid, the reduction proceeds quantitatively to the tetroxide, and admits of the estimation of vanadic acid by this method. J. V. E.

The Series Sodium Thioantimonate, Sodium Thiosulphate, and Water. A. D. DONK (*Chem. Weekblad*, 1908, 5, 767—771).—From mixtures of sodium thioantimonate and sodium thiosulphate in aqueous

solution at 0° and 30°, the following hydrated salts have been isolated: $\text{Na}_3\text{SbS}_4 \cdot 9\text{H}_2\text{O}$; $\text{Na}_3\text{SbS}_4 \cdot 9\text{H}_2\text{O} \cdot \text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$; $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

A. J. W.

Passivity of Platinum. RUDOLF RUEB (*Zeitsch. Elektrochem.*, 1908, 14, 633—634. Compare this vol., ii, 601).—Two platinum anodes in series were placed in 6% and 50% sulphuric acid respectively; after twelve hours their positions were interchanged, and this was continued for seven days; each anode lost 0.7 mg. When the anodes were not moved, they each lost 0.1 mg. In the first experiment, the cathode in the 50% acid gained 1.1 mg.; hence the coating formed in the 6% acid dissolves in the strong acid. If an invisible skin of oxide forms on an anode in the strong acid, it is therefore not the same oxide as that produced in the weak acid.

T. E.

Mineralogical Chemistry.

Coloration of Minerals. KARL SIMON (*Jahrb. Min.*, 1908, *Beil.-Bd.*, 26, 249—295).—A review is given of the literature, and experiments made on the coloration of zircon, amethyst, smoky-quartz, tourmaline, and topaz. These minerals are all decolorised when strongly heated in an atmosphere of either hydrogen or oxygen, and the temperature at which the colour is lost depends to some extent on the size of the fragments. The colour is restored on exposing the mineral to sunlight, or to the emanations of radium. The colouring matter is in each case probably inorganic, but its exact nature is unknown.

L. J. S.

Relations between Quartz, Chalcedony, and Opal. HANS LEITMEIER (*Centr. Min.*, 1908, 632—638).—The following minerals, in a state of moderately fine powder, were heated with a 50% solution of potassium hydroxide at 80° for five hours; the residue was washed with a 50% solution of potassium hydroxide before being washed with water (otherwise some of the dissolved silica is re-precipitated), and the amount of silica in solution was determined:

	Sp. gr.	Water lost at 80°.	Water lost on ignition.	Silica dissolved.
Quartz from Rauris	2.613	—	(0.22%)	7.23%
Chalcedony from Weitendorf	2.608	—	1.50	76.02
" " " dehydrated	—	—	—	53.10
Chalcedony from Faroe	2.591	0.10%	1.02	42.30
Cacholong " "	2.370	0.25	1.35	54.40
Opal (Hyalite) from Bohemia	2.177	0.34	3.04	56.63
Opal (precious), New South Wales	2.121	2.40	6.23	100.00
" " " dehydrated	—	—	—	82.34

Hot water acting for twelve hours on precious opal gelatinises the surface and dissolves 0.22%. It is concluded that chalcedony and

quartz are varieties of the same mineral species, and that chalcedony does not contain any admixed opal.

L. J. S.

The Reversible Alteration of Cryolite. FELIX CORNU (*Centr. Min.*, 1908, 546—547).—R. NACKEN (*Centr. Min.*, 1908, 38) has observed the change of monoclinic cryolite to a cubic modification at a temperature of 550—570°. The suggestion is made that this cubic modification ($\text{Na}_6\text{Al}_2\text{F}_{12}$) is isomorphous with cryolithionite ($\text{Li}_3\text{Na}_3\text{Al}_2\text{F}_{12}$) (*Abstr.*, 1904, ii, 347).

L. J. S.

Origin of the Boric Acid in the Soffioni of Tuscany. GIOVANNI D'ACHIARDI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 238—239).—A reply to Nasini (compare this vol., ii, 862).

T. H. P.

Manganese and Iron Minerals from the Crystalline Schists, of Broșteni, Roumania. VASILE C. BUTUREANU (*Ann. Sci. Univ. Jassy*, 1908, 5, 87—108).—Fifteen analyses are given of "brostenite" (*Abstr.*, 1901, ii, 26); they exhibit wide variations, indicating that the material is not homogeneous. Analyses are also given of wad, and a manganese and iron carbonate.

L. J. S.

Vorobyevite and the Chemical Structure of Beryls. WLADIMIR I. VERNADSKY (*Bull. Acad. Sci. Petersburg*, 1908, 975—976).—The author describes a beryl containing caesium, to which he gives the name *vorobyevite*. From the best published analyses, the conclusion is drawn that the beryls constitute a class of minerals by themselves. Various members of this class are known, having the composition: $p\text{-GlAl}_2\text{SiO}_{12}.qA$, where A is GfH_2SiO_4 , $\text{GfSiO}_3(t)$, Cs_2SiO_3 , Li_2SiO_3 , or Na_2SiO_3 . In beryls not containing alkali metals, the compound $2\text{GlAl}_2\text{Si}_4\text{O}_{12}(\text{GfSiO}_3)_4(\text{GfH}_2\text{SiO}_4)$ predominates. Vorobyevite contains 4.65—10.4% of a caesium compound of the type $\text{GlAl}_2\text{Si}_4\text{O}_{12}(\text{GfH}_2\text{SiO}_4)(\text{Cs}_2\text{SiO}_3)$.

T. H. P.

Analysis of the Microcline from the Pegmatites of Mesvres. PHILIPPE BARBIER (*Bull. Soc. chim.*, 1908, [iv], 3, 821—822).—The pegmatites of the Mesvres valley contain a beautiful slightly translucent, laminated, milk-white, or pink microcline. The author has analysed specimens of this mineral collected at Mesvres, and obtained the following results. An analysis by Damour of specimens of the same microcline found at Broye is given for comparison:

	SiO_2 .	Al_2O_3 .	K_2O .	Na_2O .	Total.
I. Mesvres . . .	66.12	19.11	10.54	4.12	99.93
II. „	66.13	19.09	10.62	4.12	99.96
III. Broye . . .	64.80	19.90	12.11	2.10	98.91

E. H.

Analysis of the Christianite of Simiouse. PHILIPPE BARBIER (*Bull. Soc. chim.*, 1908, [iv], 3, 822—823).—The cavities in a basalt of Mt. Simiouse, near Montbrison, contain microscopic crystals of christianite and other minerals. The christianite gave on analysis:

SiO ₂	Al ₂ O ₃	CaO.	K ₂ O.	Na ₂ O.	BaO, MgO.	Loss on ignition.	Total.
52.10	18.33	4.96	6.89	1.12	traces	16.55	99.95

This mineral thus corresponds closely with the formula
 $\text{Si}_5\text{O}_{14}\text{Al}_3(\text{K}, \text{Ca}) + 5\text{H}_2\text{O}.$

E. H.

Aloisiite: a New Hydrosilicate from the Tufa of Fort Portal (Uganda). LUIGI COLOMBA (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 233—238).—The silicate contained in the tufa of Fort Portal is of a type extremely poor in silica, and after deducting admixed calcium carbonate and gangue, has the following composition:

SiO ₂	FeO.	CaO.	MgO.	Na ₂ O.	H ₂ O.	Total.
24.52	20.56	26.50	11.08	9.96	6.95	99.57

These numbers correspond approximately with the formula $(\text{R}'\text{R}'_2)\text{SiO}_6$, where $\text{R}'\text{O}$ is CaO , FeO , MgO , and $\text{R}'_2\text{O}$ is $\text{Na}_2\text{O}, \text{H}_2\text{O}$. The author gives to this silicate the name *aloisiite*; it occurs also in an altered form in the tufa.

T. H. P.

Formation of Kaolin. OTTO HÄHNEL (*J. pr. Chem.*, 1908, [ii], 78, 280—294).—Analyses of a fresh (I), an effloresced (II), and a half-kaolinised porphyrite show that the processes of efflorescence and kaolinisation are not identical:

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO.	CaO.	K ₂ O.	Na ₂ O.	H ₂ O.
I.	72.9	15.4	2.89	—	0.41	4.40	5.11	0.5 per cent.
II.	70.98	15.50	3.21	traces	0.72	4.70	5.01	2.21 „ „
III.	75.0	18.72	1.2	—	0.2	2.31	2.78	1.03 „ „

On the other hand, the fresh decomposition products of a granite from under humus at Schierke closely resembles crude kaolin. The moors must, therefore, be credited with a kaolinising power. G. Y.

Calcium Sulphide (Oldhamite) in the Allegan Meteorite. WIRT TASSIN (*Proc. U.S. National Museum*, 1908, 34, 433—434).—Certain portions of this meteoric stone when treated with acid were noticed to evolve a considerable amount of hydrogen sulphide, and an analysis of the fine powder, freed from magnetic particles, showed the presence of 16.66% CaS in an olivine-enstatite mixture. This constituent could not be detected on a microscopical examination of the stone, and it appears to be present in a finely-divided state in the ground mass.

L. J. S.

Meteoric Chromites. WIRT TASSIN (*Proc. U.S. National Museum*, 1908, 34, 585—590).—Small quantities of chromite are present in

most meteorites; in stones and stony irons rarely as much as 3%, and usually less than 1%, whilst in irons there is generally less than 0.01%. The following nine analyses were made on material isolated from the meteorites named, and eight earlier analyses of meteoric chromites are quoted.

are quoted.	Cr ₂ O ₃ .	Al ₂ O ₃ .	Fe ₂ O ₃ .	FeO.	MgO.	SiO ₂ .
I. Admire.....	63.39	—	—	33.00	0.40	0.50
II. Mount Vernon.....	66.01	9.95	—	18.37	5.08	—
III. ".....	64.91	9.85	—	17.92	4.99	1.38
IV. Canyon Diablo.....	63.40	5.30	—	26.30	5.00	—
V. Marjashah.....	61.39	1.96	—	30.46	6.70	—
VI. Hendersonville.....	56.73	2.98	—	29.84	2.42	—
VII. Allegan.....	56.70	12.38	—	27.60	4.00	—
VIII. Admire.....	56.49	trace	10.20	29.82	trace	—
IX. Canyon Diablo.....	5.20	—	65.25	30.05	—	—

* Trace of TiO_2 .

From the Admire pallasite, I was of small, non-magnetic, jet-black grains with a brilliant lustre; and VIII, of bluish-brown, magnetic particles with a dull lustre, isolated from the metallic portion of the meteorite. II, from the metallic portion of the Mount Vernon pallasite, had the form of relatively large (1 mm. across) octahedra with black colour and brilliant metallic lustre, D 4.49; whilst III had the form of minute, rounded grains, frequently enclosed in the olivine, and was brownish-black with a resinous lustre. IV, from the Canyon Diablo iron, had the form of small octahedra and rounded grains, was jet-black with brilliant lustre, and non-magnetic; IX, from the same iron, was in rounded grains, with bluish-black colour and dull lustre, and was strongly magnetic, being really a chromiferous magnetite.

Only the first of these analyses approximates to the typical chromite formula, $\text{FeO} \cdot \text{Cr}_2\text{O}_3$; the others contain variable amounts of alumina and magnesia, but are of the type $\text{RO} \cdot \text{R}_2\text{O}_3$. L. J. S.

L. J. S.

Physiological Chemistry.

The Total Sugar of the Blood. RAPHAEL LÉPINE and BOULUD (*Compt. rend.*, 1908, 147, 226—228).—The total amount of the reducing sugar of the blood can be ascertained by hydrolysing the clot with hydrofluoric acid, and adding the amount of reducing substance thus obtained to that in the filtrate from the clot. S. B. S.

S. B. S.

The Occurrence of Proteose in Blood and Urine. L. BORCHARDT (*Zeitsch. physiol. Chem.*, 1908, 57, 305—312).—If elastin is given in the food in fairly large amounts to dogs, a proteose (hemielastin) can be detected in the blood, and this is excreted by the kidneys and found in the urine. W. D. H.

W. D. H.

Fatty Degeneration in the Blood. SAMUEL G. SHATTOCK and LEONARD S. DUDGEON (*Trans. Path. Soc. London*, 1907, 58, 227—255).—In preparations of blood corpuscles hardened in formaldehyde, and stained with Scarlet [?Scarlet R], it is contended that stained particles in the leucocytes in various cases of disease indicate the occurrence of fatty degeneration.

W. D. H.

Estimation of Catalases and Oxydases in Blood. II. WALTHER LÖB and PAUL MULZER (*Biochem. Zeitsch.*, 1908, 13, 475—495).—The rate of oxygen evolution was estimated both by the volume and pressure methods already described (this vol., ii, 999). The rate of reaction depends on the quantity of blood added. The curves representing the rate of gas evolution show with small quantities of blood a gradual rise. In the presence of larger quantities, the gradual rise is preceded by a sharper one. The velocity constant is not the same as that of the hæmase studied by Senter, and, from the complexity of the reaction, it is possible that more than one enzyme takes part in the reaction. The rate of action varies with the blood of different animals (rabbits). The action of various oxygen depolarisers was studied. Benzidine, formaldehyde, and salicylic acid are readily oxidised, but not alcohol.

S. B. S.

The Regulation of the Physico-chemical Properties of the Blood after Injection of Different Solutions. GIUSEPPE BUGLIA (*Biochem. Zeitsch.*, 1908, 13, 400—439).—Hypertonic, isotonic, and hypotonic saline and sucrose solutions were injected into dogs, and certain quantities of blood were withdrawn from the animals both before and at different intervals after the injections. Determinations were made of the conductivity and viscosity of the serum, the molecular concentration of the defibrinated blood (by lowering of freezing-point method), and the volume of the corpuscles (by the hæmatocrite). Injections of hypertonic sodium chloride solutions were found to increase the molecular concentration of the blood and conductivity of the serum over a relatively long period, whereas they caused only a very temporary dilution of the blood volume. The changes produced by hypertonic sucrose solutions were only of temporary character, and disappeared much more quickly than those produced by sodium chloride. Isotonic solutions produced but slight changes, and so also did strongly hypotonic solutions, when the volume of the injected liquid and the rate of injection were not great. If, however, the quantity of the fluid and the rate of injection is sufficiently great to produce marked changes in the blood elements, then the physico-chemical changes are demonstrable.

S. B. S.

The Influence of Alkaline Ferro- and Ferri-cyanides on Blood-coagulation. J. LARGUIER DES BANCLES (*Compt. rend.*, 1908, 147, 266—268).—Both potassium ferro- and ferri-cyanides have a marked inhibitory action on the coagulation of the blood and of fibrinogen solutions, the latter acting more powerfully than the former, although it gives no precipitate with soluble calcium salts, whereas the

former does. It is suggested that multivalent negative ions inhibit the coagulation of negative colloids.

S. B. S.

Hæmolytic Poisons, especially Bile Salts and Soaps.

F. NEUFELD and HÂNDEL (*Chem. Zentr.*, 1908, ii, 891—892; from *Arch. Kais. Gesundh. Amt.*, 1908, 28, 572—584).—This is a study of the corpuscle-dissolving action of sodium taurocholate, soaps, and potassium hydroxide in comparison with other blood poisons, also of the action of hæmolytic agents on lipoids and proteins, so that their action on the cell-membrane and their destructive action on the whole corpuscle may be distinguished. Lecithin is soluble in a 10% solution of sodium taurocholate, but not in normal potassium hydroxide, or in a 1% solution of an oleate. Cholesterol crystals are soluble in none of these solutions, nor in sapotoxin. The taurocholate dissolves protein. In emulsions of olive oil in physiological salt solution, partly with lecithin and partly with egg-white, the addition of sodium taurocholate produces the liberation of fat, and the solution of lecithin and protein; the other materials used do not produce this effect.

In high concentrations, sodium taurocholate produces in the blood of sheep, goat, and ox an inhibition of hæmolysis, which in lower concentration rises as the concentration increases to an optimum. These three sorts of blood are peculiar in that they are refractory to pure cobra venom; the venom acts hæmolytically in the presence of lecithin. Other kinds of blood (guinea-pig, horse, hen) never show any inhibition unless physiological salt solution is replaced by an isotonic solution of sucrose.

Sodium taurocholate and a series of other hæmolytic agents are capable of uniting with complement, but this is not the case with all blood poisons; it depends on what constituent of the stroma or serum the poison is united to. In the case of sapotoxin, there is, for instance, no union with complement. The destruction of pneumococci by bile salts is also described, so also is "phagocytosis by emulsified fat droplets and the influence of specific anti-sera thereon."

W. D. H.

Hæmolysis. LEO VON LIEBERMANN (*Biochem. Zeitsch.*, 1908, 13, 363—364. Compare Abstr., 1907, ii, 973).—In order to produce hæmolysis of the corpuscles of pig's blood, 0.05 c.c. of pure oleic acid, and not oleic acid emulsion, as stated in the previous paper, should be added to 10 c.c. of pig's serum. There is also an error in the same place, as to the order in which different substances should be added to produce hæmolysis.

S. B. S.

Bactericidal Action of Normal Serum. ROBERT MUIR and CARL HAMILTON BROWNING (*J. Pathol. Bacteriol.*, 1908, 13, 76—91).—A distinction is drawn between absorption of complement by bacteria and the bactericidal effect which may follow. Treatment of a normal serum with increasing amounts of a dead emulsion of a bacterium usually produces first a diminution of its bactericidal action on that bacterium, then for that on other bacteria, and finally a diminution of the hæmolytic complement. This shows there is in serum a substance

(bacteriophilic complement) with an affinity for bacteria in general. If the bactericidal action has been reduced as indicated above, it can be more than restored by adding a little of the homologous immune substance.

W. D. H.

Digestion of Fat. III. S. A. LEVITES (*Zeitsch. physiol. Chem.*, 1908, 57, 46—48. Compare Abstr., 1907, ii, 891).—The glycerol constituent of fat is rapidly absorbed. Absorption begins in the duodenum, and is completed in the neighbourhood of the cæcum. In the estimation of glycerol in the intestinal contents, the method of Shukoff and Schestakoff, which depends on the solubility of glycerol in acetone, gives results which are too high. Lewkowitsch's acetin method is better.

W. D. H.

Nutritive Value of Plant Amides. ERNST SCHULZE (*Zeitsch. physiol. Chem.*, 1908, 57, 67—73).—A critical review of the difference of opinion which has arisen between Lehmann, on the one hand, and Morgen and Henriques and Hansen, on the other, with reference to the nutritive value of asparagine and similar amides.

W. D. H.

Ferments of Nuclein Metabolism. ALFRED SCHITTENHELM (*Zeitsch. physiol. Chem.*, 1908, 57, 21—27. Compare Abstr., 1907, ii, 109, 564).—Previous work by the author (and by W. Jones) has shown that the formation of uric acid from nuclein is due to a succession of ferment actions. It is now shown that these ferments can be precipitated from organ-extracts by alcohol or by ammonium sulphate, and these ferments, when acting on pure guanine, resolve it quantitatively into uric acid; under favourable conditions, the time may be as short as one to two hours. Bacterial action, which some authors lay stress on, is not the cause of the change.

W. D. H.

Chemistry of Digestion. XXVI. The Behaviour of Different Proteins in the Stomach and Upper Duodenum of the Dog. E. S. LONDON and W. W. POLOWZOWA (*Zeitsch. physiol. Chem.*, 1908, 57, 113—130. Compare this vol., ii, 870).—Various proteins of animal and vegetable origin are not absorbed in the stomach. Proteins from serum and egg-white are difficult of digestion in the stomach, but most others are rendered soluble to the extent of 78%, the proportion between proteoses, peptones, and residual substances being 59.3, 32.9, 7.8. The juices in the first part of the duodenum act immediately on the soluble products of gastric digestion, resolving them into ultimate cleavage products, whereas the undissolved substances in the chyme are not attacked for some time. The first 6 or 7 centimetres of the duodenum absorb about 6% of the protein.

W. D. H.

Absorption of Protein. KORNÉL VON KÖRÖSY (*Zeitsch. physiol. Chem.*, 1908, 57, 267—287).—The view is advanced that the cleavage products of protein digestion cannot be detected in the circulating blood; the percentage of protein in the blood is, however, increased, although whether protein synthesis occurs in the intestinal wall or in

the blood itself is unsettled. The non-coagulable nitrogen in the blood is not increased, as stated by Cathcart and Leathes. W. D. H.

Muscular Activity and Protein Metabolism. PHILIP A. SHAFFER (*Amer. J. Physiol.*, 1908, 22, 445—455).—The experiments made on two men, and given with full details, showed a rise of total nitrogen excreted in one, and a fall in the other, with muscular work. Work within physiological limits has *per se* no effect on protein metabolism as indicated by the nitrogen and sulphur of the urine. The excretion of uric acid, urea, ammonia, and creatinine is wholly unaffected.

W. D. H.

The Value of Protein Cleavage Products in the Animal Organism. VII. EMIL ABDERHALDEN and JOSEF OLINGER (*Zeitsch. physiol. Chem.*, 1908, 57, 74—79. Compare this vol., ii, 51).—The present experiments on a dog fed with the final cleavage products of casein and meat, confirm results recorded previously that such substances are capable of maintaining weight and nitrogenous equilibrium.

W. D. H.

The Influence of Nitrogen-free Sources of Energy on the Rate of Protein Decomposition by the Organism. W. FALTA and A. GIGON (*Biochem. Zeitsch.*, 1908, 13, 267—273).—Dogs of similar size were fed on a standard diet for a time, and then allowed to fast for various periods. They were then given diets consisting, in one set of experiments, of meat alone; in another, of a mixture of meat and levulose; in another, of meat and inosite, and in a fourth set, of meat and alcohol. It was found that the longer the period of fasting before administration of the meal the more rapid the protein decomposition (as estimated by the nitrogen excreted), and the less the protein-sparing action of the carbohydrate when this formed part of the food. The addition of inosite only slows the rate of protein decomposition during the first twelve hours. The time of the fast before the meal has no influence on the action of inosite. Alcohol accelerates the rate of protein decomposition. There is a tendency in the organism to replenish the glycogen stores which had become depleted by fasting, and this is done by obtaining the carbohydrate entirely from the protein when no other glycogen-former is ingested; hence, after fasting, the rapid degradation of protein. The carbohydrate store in the organism has therefore great influence on the time of decomposition. Experiments were also carried out on the effect of superposing various extra diets on a standard diet. It was found that the composition of the standard diet exerts considerable influence on the time of decomposition of the superposed diets.

S. B. S.

The Influence of Nitrogen-free Sources of Energy on the Rate of Protein Decomposition by the Organism. G. A. PARI (*Biochem. Zeitsch.*, 1908, 13, 274—280).—The experiments were similar in character to those of Faltz and Gigon (see preceding abstract). The effects of the addition of carbohydrates other than

lævulose and of fat, on the rate of protein decomposition after a fast, were investigated. It was found that after a long fast, sucrose no longer has the power of slowing the rate of protein decomposition. Fat and β -hydroxybutyric acid, which are not glycogen formers, do not lose, however, their power to retard the rate of decomposition.

S. B. S.

The Influence of the Thyroid Gland on the Rate of Decomposition. G. A. PARI (*Biochem. Zeitsch.*, 1908, 13, 281—284).—Eppinger, Falta, and Rudinger have shown that in dogs without thyroid glands, the protein metabolised during fasting is less, and can be but little diminished by carbohydrates or fat, in contrast to what happens in the normal starving animal. The latter fact is attributed, from other factors discovered, to the depression of carbohydrate metabolism in animals deprived of the thyroid. To throw further light on this matter, experiments similar to those recorded in the preceding abstracts were carried out with animals deprived of the thyroid, which received meals of meat, with or without addition of other food-stuffs, after a period of fasting, and the rate of protein decomposition was determined by estimating the rate of nitrogen excretion. In the normal animal, the influence of carbohydrates on the retardation of the decomposition of protein is diminished by longer periods of fasting. In thyroidectomised animals, long periods of fasting do not diminish this delaying influence to anything like the same extent.

S. B. S.

Influence of Thyroidectomy and Thyroid Feeding on Intermediary Metabolism. FRANK P. UNDERHILL and TADASU SAIKI (*J. Biol. Chem.*, 1908, 5, 225—242).—After complete thyroidectomy and parathyroidectomy in dogs, the urinary ammonia is increased beyond what is observed in inanition. The nitrogen in the form of creatinine, purine, and allantoin is unaltered, but creatine is found in the urine. These dogs are incapable of utilising dextrose introduced subcutaneously to anything like the same degree as normal dogs can. The loss of the glands may thus cause a change in gaseous metabolism similar to that seen in cretinism. If normal dogs are fed on thyroid, the urinary nitrogen is increased; there is also a larger output of purine-nitrogen, and a low output of phosphorus. There is, however, but little change in the inter-relation of the urinary nitrogenous constituents.

There is no choline in the blood after thyroidectomy. W. D. H.

The Utilisation of the Energy of Provender as Influenced by the Temperature of the Surroundings, and the Nutritional Condition of Rabbits. W. USTJANZEFF and G. BOGAJEWSKY (*Biochem. Zeitsch.*, 1908, 13, 365—399).—Rabbits were fed for a preliminary period on a given diet which was insufficient for the energy needs of the organism. The carbon and nitrogen content, and also the calorific value of this diet and of the excreta, were estimated, and from these data, the loss from the body of fat and protein calculated, and also the calorific value of this loss. The same animal was then given the like diet with the addition of hay, and

the same data obtained as in the preliminary period. From the two sets of data, the physiological food value of the hay was calculated. Experiments were carried out with animals in varying conditions of nutrition, and with the surrounding atmosphere at varying temperatures (0° , 20° , and 30°). It was found that Rübner's law of isodynamic replacement holds good if hay is used as provender, when the physiologically available energy of the latter is only about half of the total energy needs of the animal, and when the animal is kept in an atmosphere of low temperature.

S. B. S.

Secretin. OTTO VON FÜRTH and CARL SCHWARZ (*Pflügers Archiv*, 1908, 124, 427—446).—Extracts of intestinal mucous membrane, prepared according to the method of Bayliss and Starling, contain choline. Choline stimulates pancreatic activity, and this action is antagonised by atropine. The secretin of Bayliss and Starling is a mixture of several substances, among which choline occurs. The effect of secretin is not abolished, but only lessened, by atropine.

W. D. H.

Oxidation Processes in Echinoderm Eggs. OTTO WARBURG (*Zeitsch. physiol. Chem.*, 1908, 57, 1—16).—The research deals with the respiratory process, measured by the amount of oxygen used, in the eggs of *Arbacia pustulosa*. The figures given show (1) that the amount of oxygen used is from six to seven times greater in fertilised than in unfertilised eggs; (2) this is not mainly used in nuclear division, for at the stage when thirty-two cells are present, the use of oxygen is not proportionately increased from the stage when eight cells are present; (3) moreover, if the continuance of cell division is hindered by hypertonic sea-water, the amount of oxygen used is not markedly altered; (4) an egg-cell breathes 500 (± 100) times more vigorously than a sperm cell; (5) hypertonic solutions increase the use of oxygen in unfertilised eggs ten-fold; (6) a transference from hypertonic to normal sea-water also leads to an increase, but not such a marked one; (7) the temperature-coefficient shows the process to be a chemical one.

W. D. H.

A Mono-amino-diphosphatide in Egg-Yolk. HUGH MACLEAN (*Zeitsch. physiol. Chem.*, 1908, 57, 304).—A mono-amino-diphosphatide (P : N = 2 : 1) analogous to Erlandsen's cuorin, which he separated from heart muscle, can be separated from egg-yolk.

W. D. H.

A Globulin from the Egg-Yolk of *Squalus acanthias*. CARL L. ALSEBERG and E. D. CLARK (*J. Biol. Chem.*, 1908, 5, 243—246).—The egg-yolk of the spiny dogfish contains no vitellin, but in its place, a globulin (or a mixture of globulins) which does not contain phosphorus, and, probably, iron is also absent.

W. D. H.

The Assimilation of Phosphorus and Calcium during the Embryonic Life of the Chick. EM. CARPIAUX (*Bull. Acad. roy. Belg.*, 1908, 283—295).—Analyses were made of the inorganic and lecithin-phosphorus and of the calcium in eggs in different stages of develop-

ment. It was found that the chick during embryonic life uses chiefly the phosphorus in the egg itself, and if it utilises that of the shell at all, it does so only to a very limited extent. The shell furnishes four-fifths of the calcium necessary for the formation of the chick, and towards the end of the incubation period considerable use is made of this calcium. At this period, destruction of the lecithin takes place, which proceeds *pari passu* with the utilisation of the lime.

S. B. S.

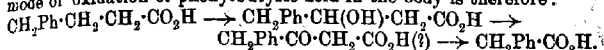
The Peroxydases of Animal Tissues. FR. BATTELLI and MILZ. L. STERN (*Biochem. Zeitsch.*, 1908, 13, 44—88).—Tissue extracts possess the capacity of increasing the rate of oxidation of hydriodic acid by means of hydrogen peroxide. The reaction is manifested when carried out in the presence of starch. In the cases of most tissues, the reaction is masked by the presence of a catalase, which causes a too rapid destruction of the peroxide. If, however, ethyl hydroperoxide is employed instead of hydrogen peroxide, the reaction can, in most cases, be demonstrated, for the catalase of the tissue does not act on this substance, which possesses a similar peroxydase reaction to hydrogen peroxide. Even when the former peroxide is employed, the results are uncertain, and this fact is probably due to the presence of the substances in the tissues, which react chemically with iodine and thus mask the iodine-starch reaction. A more suitable method of investigating the peroxide reaction of the tissues consists in estimating the amount of carbon dioxide evolved when tissue extracts are treated with ethyl hydroperoxide or hydrogen peroxide and formic acid. The oxidation which then takes place, can occur in acid solutions, under which condition the catalase action on hydrogen peroxide is considerably depressed. The following tissues were investigated, and are enumerated in order of the magnitude of their peroxydase content: liver, kidneys, spleen, lungs, pancreas, lymph glands, ox-muscle, brain, testicles, dog-muscle, thymus, suprarenals, thyroid, rabbits muscle. Blood is also rich in peroxydase. The optimum temperature of reaction in most cases is 38—40°, and the ferment is destroyed at 66° in neutral media and at 55° in acid or alkaline media. In the case of the blood, however, the reaction is much more energetic at 55—60°, and is in other respects anomalous. It is stated to contain a so-called pseudo-peroxydase in the pigment. The formaldehyde is also oxidised under the same conditions as formic acid, but not so energetically. Higher fatty acids, which *in vivo* are readily oxidised, do not cause evolution of carbon dioxide under the same conditions as formic acid does. For this reason, it is not yet possible to determine the exact biological functions of peroxydases.

S. B. S.

Mode of Oxidation of Phenyl Derivatives of Fatty Acids in the Animal Organism. HENRY D. DAKIN (*J. Biol. Chem.*, 1908, 5, 173—186).—The subcutaneous injection of sodium phenylbutyrate results in the excretion of phenaceturic acid and a small quantity of β -hydroxy- γ -phenylbutyric acid. No phenylacetone or phenylacetoacetic acid could be found.

If β -hydroxy- γ -phenylbutyric acid is given in the same way, phenaceturic acid is again excreted, and phenylacetone could not be detected.

Administration of phenylacetone results in the excretion of hippuric acid, phenylacetic acid not being formed. The probable mode of oxidation of phenylbutyric acid in the body is therefore:



Phenylacetic acid is excreted as phenacetic acid. The intermediate formation of ketones is probably confined to phenylbutyric and phenylpropionic acids, and is not a general reaction. In normal metabolism, probably only part of the butyric and phenylpropionic acids passes through the stage of acetone and acetophenone respectively.

Phenylisocrotonic acid is excreted as phenacetic acid.

β -Dihydroxyphenylbutyric acid is excreted as hippuric acid and a little β -hydroxyphenylbutyrolactone. Mandelic acid is not formed. Dihydroxyphenylbutyric acid therefore does not undergo β -oxidation, but oxidation takes place at the γ -carbon atom. Dihydroxyphenylbutyric acid is not a product of the catabolism of phenylbutyric acid.

Phenylacetone is readily identified by conversion into its *p*-nitrophenylhydrazone, which crystallises from alcohol or pyridine in sparingly soluble rosettes of platelets, melting at 145–145.5°.

W. D. H.

Chemistry of the Brains of Birds and Fishes. ALFRED ARGIRIS (*Zeitsch. physiol. Chem.*, 1908, 57, 288–295).—Cerebron from human brain yields 21.83% of galactose; that from birds' brain, 21.75%. A crystalline substance corresponding with Thudichum's sphingosin was also obtained. The birds employed were hens and ducks. Fishes' (cod) brain yielded cholesterol, lecithin, jecorin, and so-called protagon.

W. D. H.

The Chemical Composition of Peripheral Nerves. FRITZ FALE (*Biochem. Zeitsch.*, 1908, 13, 153–172).—Both medullated and non-medullated nerves were investigated (sciatic of man, and splenic nerves of ox). The dried medullated nerves were extracted consecutively with benzene, acetone, and ether; the ether-soluble fraction was treated with alcohol, and to the filtrate from the precipitate thus produced, ammoniacal lead acetate in alcohol was added and, after separation of the precipitate and excess of precipitant, alcoholic cadmium chloride solution. By means of the various solvents and precipitants, a series of fractions was obtained, which were partly identified with known substances. The benzene-soluble fraction consisted chiefly of ordinary fats; the acetone extract contained chiefly cholesterol (m. p. 145°); from the ethereal extract a cerebroside separated, and by the precipitation of this extract with alcohol, the cephalins. Ammoniacal lead acetate precipitated another product belonging to the group of cephalins (m. p. 174°). The cerebroside was identical with the cerebrone of Thierfelder (m. p. 209°). The cephalin (m. p. 174°) had the chemical composition of a mono-amino-monophosphatide; it could not be identified with the cephalins already described and obtained from the brain. Only a

small quantity of cadmium chloride precipitate soluble in cold benzene was obtained, and the amount of lecithin appeared therefore to be small and to be accompanied by other phosphatides, which have not yet been identified. A quantitative separation of the various constituents of medullated and non-medullated nerves showed that the former contained 25% cholesterol, 12.4% cephalins, 18.3% cerebrosides, and 2.9% lecithin, whereas the latter contained 47% cholesterol, 23.7% cephalins, 6% cerebrosides, and 9.8% lecithin.

S. B. S.

Weigert's Method of Staining Medullated Nerve Fibres. J. LORRAIN SMITH, W. MAIR, and JOCELYN F. THORPE (*J. Pathol. Bacteriol.*, 1908, 13, 14—27).—Weigert's method of hæmatoxylin staining, after treatment with potassium dichromate, depends, as osmic acid staining does, on the presence of an unsaturated grouping in the fatty matter present. Cholesterol by itself gives a negative result, although it contains an unsaturated grouping, but it forms myelin figures in contact with soap. These and the crystals described in tissues by C. P. White (composed of cholesterol and fatty acid) stain slowly by the Weigert method. Some results obtained with lecithin are attributed to cholesterol as an impurity in the lecithin used.

W. D. H.

Action of Curare and Physostigmine [Eserine] on Nerve-endings. Action of Barium Chloride on Bird's Muscle. CHARLES WALLIS EDMUND and GEORGE B. ROTH (*Amer. J. Physiol.*, 1908, 23, 28—45, 46—47).—The tonic contraction produced by nicotine in muscles is counteracted by curare; this antagonism is less marked if the nerves are cut, and, as a rule, this lessening of the antagonism coincides in data with the degeneration of the nerve-ending. This may be because denervated muscle has an increased sensitiveness towards nicotine, so that curare is unable to displace it. This view is upheld, and so curare is considered to act, not on the nerve-ending as usually taught, but on the muscle, or on some substance in the muscle of the nature of Langley's receptive substance. As a support to this view, experiments with eserine are recorded. This alkaloid acts in the same way as nicotine, only more slowly. It acts more quickly on denervated muscle, which, therefore, has an increased sensitiveness to it. Curare neutralises the effect whether the muscles are denervated or not. The tonic contraction produced by barium chloride is, however, not antagonised by curare under any circumstances.

W. D. H.

Chemico-physical Investigation of the Crystalline Lens. FILIPPO BOTTAZZI and NOE SCALINCI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 153—159).—The aqueous humour of the eye contains normally a very small proportion of protein, which is coagulable by heat, but has not the characters of the lenticular protein, and probably has its origin in the blood or lymph. The vitreous body resembles a sponge, the net-work of which consists of protein substances coagulable by heat, and contracts during coagulation, expressing the liquid contained

the alveoli. This liquid contains a small quantity of a substance precipitable by acetic acid and insoluble in excess of the acid, and is possibly identical with the coagulable protein found in the aqueous humour. Neglecting these small amounts of proteins, the ocular liquids may be regarded as aqueous solutions of crystalloids, mainly of sodium chloride. With regard to their chemical reaction, the ocular liquids behave like a solution of sodium hydrogen carbonate containing sufficient carbon dioxide to render it neutral to phenolphthalein. Excess of carbon dioxide in the ocular liquids renders them acid to phenolphthalein, and may result in the precipitation of the protein as alkali-protein and in the production of superficial opacity of the crystalline lens.

T. H. P.

Amount of Choline in the Lecithin of Heart Muscle. HUGH MACLEAN (*Zeitsch. physiol. Chem.*, 1908, 57, 296—303).—The lecithin of heart muscle when decomposed in aqueous or alcoholic solution yields, on the average, not more than 52% of its nitrogen in the form of choline. Whether a second nitrogenous product is also present is to be investigated.

W. D. H.

Hydrolysis of Chicken Flesh. THOMAS B. OSBORNE and FREDERICK W. HEYL (*Amer. J. Physiol.*, 1908, 22, 433—439).—The entire muscle was used, the substances soluble in water, alcohol, and ether having been removed. The result of hydrolysis shows that the amino-acids occur in different proportions from those given previously by Abderhalden and Sasaki and by Hart in relation to the syntonin of ox flesh. It is, however, doubtful whether the analyses can be fairly compared, especially as the sum total of the products in one case is 50%, and in the other 47%, of the material employed. The most striking feature in the hydrolysis of chicken muscle is the high yield of lysine.

W. D. H.

The Degradation of Acetoacetic Acid in the Animal Body. II. GUSTAV EMBDEN and LOUIS MICHAUD (*Biochem. Zeitsch.*, 1908, 13, 262—266).—It has been already shown that the liver tissue possesses the property of destroying acetoacetic acid. As the latter is excreted in cases of diabetes, experiments were carried out to determine whether this is due to increased production, or to the loss of capacity of the liver in diabetic individuals to destroy the acid, which is probably a normal intermediary metabolism product. For this purpose, experiments were made with the liver of dogs which had been rendered diabetic by the extirpation of the pancreas. Control experiments were carried out with the liver tissue of similar normal animals. It was found that the capacity for destroying acetoacetic acid was the same in the liver of the diabetic as in that of normal animals. The muscular tissue of diabetic animals also retained its property of destroying the acid.

S. B. S.

The Behaviour of Creatine in Autolysis. A. ROTHMANN (*Zeitsch. physiol. Chem.*, 1908, 57, 131—142. Compare Stangassinger, this vol., ii, 515).—According to Gottlieb and Stangassinger (Abstr.,

1907, ii, 637), creatine and creatinine are destroyed or altered by ferment action during autolysis. Mellanby (this vol., ii, 308) finds, on the other hand, that this is not the case if bacteria are excluded. The present experiments undertaken with aseptic and antiseptic precautions confirm the results of the first-named authors.

W. D. H.

The Action of Arsenic on Autolysis. LEO HESS and PAUL SAXL (*Chem. Zentr.*, 1908, ii, 338; from *Zeitsch. exp. Path. Ther.*, 1908, 5, 89—93).—The action of arsenic, even when highly dilute, is inhibitory towards the autolytic changes in organs (liver). This is designated negative catalysis.

W. D. H.

The Occurrence of Choline in Thymus, Spleen, and Lymph Glands. CARL SCHWARZ and R. LEDERER (*Pflüger's Archiv*, 1908, 124, 353—360).—The occurrence of choline in extracts of thymus spleen, and lymph glands is proved by chemical and physiological tests. The effect that extracts of these organs have in lowering blood pressure is attributed mainly to choline. Other depressor substances present were not chemically identified, but the suggestion is made that they may belong to the group of histones.

W. D. H.

The Depressor Substance in the Thyroid. OTTO VON FÜRST and CARL SCHWARZ (*Pflüger's Archiv*, 1908, 124, 361—368).—The substance present in thyroid extracts which depresses arterial pressure is identified as choline.

W. D. H.

The Chemistry of Amyloid Degeneration. OLAV HANSEN (*Biochem. Zeitsch.*, 1908, 13, 185—197).—The amyloid tissue (the "sago granules" of the spleen) was separated from the surrounding tissue by a mechanical method. The product obtained in this way was, when dried, a yellow or brown powder, which gave the usual blue coloration with iodine after addition of sulphuric acid, and the ordinary protein reactions. The C:N ratio in three different preparations was constant (approximately 4); these contained no oxidised sulphur, and consequently no conjugated sulphuric acid, and the observation of previous observers, who noted the presence of chondroitin sulphuric acid, appears to be incorrect. The amyloid tissue is, in general, very resistant to the action of proteolytic ferments; it is changed, however, by the action of pepsin and hydrochloric acid into a product which is soluble in dilute ammonia, but which no longer gives the iodine reaction. The capacity to give this reaction is also lost on treatment of the tissue with alkalis.

Although the mechanically separated amyloid tissue itself contained no oxidised sulphur, the organs from which it had been obtained did. It seems probable that the chondroitin sulphuric acid or a similar substance is formed at the same time as the amyloid tissue. No other marked differences between amyloid and normal tissue were ascertained.

S. B. S.

So-called Fatty Degeneration of the Suprarenal. CHARLES POWELL WHITE (*J. Pathol. Bacteriol.*, 1908, 13, 11—13).—The

presence of fats and lipoids in the suprarenal cortex is a physiological condition. The cortex is believed to be engaged in the secretion of cholesterol, lecithin, and fats, and this has some connexion with the regulation of growth and development. W. D. H.

Silicic Acid in Whartonian Jelly. FRANZ FRAUENBERGER (*Zeitsch. physiol. Chem.*, 1908, 57, 17—20).—The amount of silicic acid in the Whartonian jelly of the human umbilical cord is less than Schulz stated it to be. According to Schulz, the ash contains 0.6% of the acid; the figure should be 0.028. W. D. H.

The Action of Electrolytes on the Rhythmic Movements of Medusæ. I. Action of the Salts of Sea-water. ALBRECHT BETHE (*Pflüger's Archiv.*, 1908, 124, 541—578).—An investigation of the various salts, alone and in combination, on the movements of jelly fish was carried out on the lines of the well-known work of Loeb and others. The relative importance of the various ions is discussed. Thus sodium chloride by itself first stimulates and then paralyses the movements; this action is reversible; potassium stimulates the rhythm; magnesium paralyses it; the paralysing action of calcium only occurs when it is highly concentrated. W. D. H.

The Circulation of the Bile. GUSTAV BAYER (*Biochem. Zeitsch.*, 1908, 13, 215—233).—Although the bile salts are strongly toxic to individual organs, they are being continually resorbed from the intestine and entering the circulation. They then exert no toxic effect, even in cases of jaundice. This is due to the fact that the salts are changed in some manner by the serum proteins, and are thereby deprived of their toxic character. This was confirmed by investigating the toxic action of bile salts, when alone and when mixed with serum, on muscular tissue, the central nervous system, the circulation, and also the hæmolytic action. The change effected by serum on the bile salts which deprives them of their toxic character is probably of a physical nature ("colloidal envelopment"). Serum which has been previously warmed acts more efficiently in this respect than unwarmed serum. This action between serum proteins and bile salts produces a disappearance of the complement, a fact which was ascertained by allowing red blood corpuscles to act on amboceptor and complement containing sera in the presence of bile salts which had themselves been deprived of their hæmolytic action by means of an inactive serum. The change in the bile salt molecule wrought by the serum renders excretion difficult, and a bile salt thus changed will not readily dialyse. The liver cells exert a great affinity for the bile salts, and the fixation of cholates by these cells can be demonstrated *in vitro*. S. B. S.

Bile Hæmolysis. III. The Reasons of the Increased Rate of Bile Hæmolysis in Concentrated Salt Solutions. GUSTAV BAYER (*Biochem. Zeitsch.*, 1908, 13, 234—242).—It was shown that bile salts diminish the surface tension of salt solutions to a greater extent than they do that of pure water. Substances which lower the surface

tension of a solution tend to concentrate on the surface. The bile salts tend in emulsions of blood corpuscles to collect round the erythrocytes, and the more rapidly the more concentrated the salt solution used as their solvent. In concentrated salt solutions, there is a more rapid entry therefore of the bile salts (which bring about hæmolysis by their action on the lipoids) than in water. S. B. S.

The Influence of Tolyene-2:4-diamine on the Secretion of Cholesterol in the Bile. CHASOBURŌ KUSUMOTO (*Biochem. Zeitsch.*, 1908, 13, 354—362).—The polycholia, following administration of tolylene-2:4-diamine, is stated to be due to the destruction of blood corpuscles; thus supplying material for bile pigment which is derived from hæmoglobin. As the corpuscles also contain cholesterol, it might be expected, therefore, that administration of tolylene-2:4-diamine would also lead to an increased elimination of this substance in the bile. This the author has experimentally shown to be the case. The experiments were carried out on dogs with biliary fistula. S. B. S.

Hæmolytic Factors in Milk. JANET E. LANE-CLAYTON (*J. Pathol. Bacteriol.*, 1908, 13, 34—37).—Milk, when fresh, contains both complement and amoceptor in about 1/10 the strength of that in serum. In order to obtain hæmolysis, "ox-colloid" must be added; this confirms Bordet's views: "Ox-colloid" is prepared by heating ox-serum to 56° for thirty minutes, and then removing the amoceptor by adding about twice the amount of corpuscles which it can sensitize. The mixture is incubated for one hour, and centrifuged; the supernatant fluid is "ox-colloid."

On adding blood-corpuscles to milk, the cream picks them up and carries them to the top. This red plug does not occur if the milk has been previously heated for a few minutes to 70°, and this circumstance may be used to detect heated or pasteurised milk. W. D. H.

The Degree of Acidity of Urine. ADOLF JOLLES (*Biochem. Zeitsch.*, 1908, 13, 177—184).—The concentration of the hydrogen ions was estimated by determining the rate of hydrolysis of sucrose caused by it at various temperatures. At higher temperatures, for example, at 90°, the results were complicated by the decomposition of the urea and the formation of ammonium carbonate. For this reason, the urea was estimated in the urine both before and after the process of inversion, as also the acidity (by titration) and the mono- and di-phosphates. The results indicated that the concentration of hydrogen ions is very small, and corresponds very closely with that due to monosodium phosphate present in the urine. Owing to the decomposition of urea, trustworthy results by sugar catalysis are only obtainable when the temperature of the experiment does not exceed 37°. S. B. S.

The Rate of Elimination of Chloroform. D. NOËL PATON (*Proc. Roy. Soc. Edinburgh*, 1908, 28, 472—496); D. NOËL PATON and DOROTHY E. LINDSAY (*ibid.*, 497—502).—The experiments were performed on dogs and rabbits; the blood and tissues, as well as the excreta,

were examined, and the analytical results given in detail. It was found that when chloroform is given by the respiratory passages, it is first dissolved in the blood, and thus acts on the nerve-centres, excess being rapidly eliminated. If it is subcutaneously administered, and still more if given by the stomach, the assumption is slow, more stable compounds are formed, and elimination is consequently delayed. The drug has thus more time to produce a slow toxic effect upon the protoplasm of the tissues. The onset of late chloroform poisoning after anaesthesia is due to delayed elimination brought about by unusually firm fixation or by respiratory deficiency. W. D. H.

Excretion of Creatinine in Man. C. J. C. VAN HOOGENHUYZE and H. VERPLOEGH (*Zeitsch. physiol. Chem.*, 1908, 57, 161—266. Compare Abstr., 1906, ii, 186).—An extensive series of estimations of creatinine are recorded in health and disease, which confirm, in the main, the conclusions drawn by Folin and other workers. Mellanby's view, that creatinine is converted into creatine in the liver, is confirmed, as is also the statement of Gottlieb and Stangassinger that substances (probably ferments) occur in the liver and other organs which transform creatine into creatinine. If the liver is largely destroyed (for instance, by cancer), large amounts of creatine instead of creatinine pass into the urine. The lowering of hepatic activity will explain the presence of creatine in the urine in fever and hunger. In the healthy tissues and blood, creatinine is never found, as it is removed so quickly by the kidneys. W. D. H.

Excretion of Creatine and Creatinine in Health and Disease. PHILIP SHAFFER (*Amer. J. Physiol.*, 1908, 23, 1—23).—Normally, from 7 to 11 mg. of creatinine nitrogen are excreted per kilo of body-weight. This is constant from day to day and from hour to hour; it is not influenced by the volume of the urine, or by the total nitrogen excreted. This creatinine coefficient is parallel to the muscular efficiency of the individual, and in many diseases it is lowered. Creatinine is not an index of total endogenous protein catabolism, but of some special process of normal muscular metabolism. Mellanby's view that the liver is the seat of its formation, whence it reaches the muscle, is dissented from. The creatinine excretion is slightly increased in acute fevers; here it is not parallel to muscular efficiency. Unless creatine occurs in the food, it is absent from normal urine; it may be excreted in acute fevers, in the acute stages of exophthalmic goitre, by women during involution of the uterus, and in other conditions in which there is a rapid loss of muscle protein. W. D. H.

Cystinuria with Diamines. FRANCIS H. THIELE (*Trans. Path. Soc.*, 1907, 58, 255—263).—A case of cystinuria is recorded; tyrosine and other mono-amino-acids were absent from the urine. The urine contained cystine, and, in addition, excess of neutral sulphur, and also cadaverine; putrescine was present in the faeces. All the cystine was apparently derived from tissue catabolism, since starvation, changes of diet, and administration of cystine made no difference in the amount

excreted. The conclusion is drawn that the tissues were deficient in a ferment which normally removes sulphur from thio-amino-compounds.

W. D. H.

Diuresis. XV. Excretion of Sodium Chloride in Phloridzin Diabetes. JOH. BIBERFELD (*Pflüger's Archiv*, 1908, 124, 532—540. Compare Abstr., 1906, ii, 564).—Polemical. A reply to Loewy and Neubauer on their criticisms of the author's previous work.

W. D. H.

Morphine Diabetes. W. SPITTA (*Chem. Zentr.*, 1908, ii, 343, from *Zeitsch. exp. Path. Ther.*, 1908, 5, 94—104).—The reducing substance often found in the urine of those dosed with morphine is probably lævulose, although it is stated to be optically inactive. Dextrose, glycuronic acid, and pentose are absent.

W. D. H.

Gout. HEINRICH KIONKA (*Chem. Zentr.*, 1908, ii, 342; from *Zeitsch. exp. Path. Ther.*, 1908, 5, 131—141, 142—146).—After the administration of uric urid, glycine can be detected in the blood. A crystalline derivative of it, naphthalenesulphonylglycine, was prepared, which showed all the crystallographic constants of the same substance prepared synthetically. A crystallographic description is also given of the corresponding derivatives of other amino-acids (leucine, alanine, &c.).

Feebly alkaline solutions of glycine, alanine, leucine, and allantoin catalytically accelerate the precipitation of acid urates from solutions of uric acid. The presence of such substances in gout is therefore harmful.

W. D. H.

High Temperatures and Heat Stroke. HARVEY SUTTON (*J. Pathol. Bacteriol.*, 1908, 13, 62—73).—In the human subject, the rise of internal temperature, due to exposure to high external temperature, is accompanied by a marked rise in total respiratory exchange and a rise in the respiratory quotient; this probably points to a greatly increased combustion of carbohydrates.

W. D. H.

Crystals in Tumours. CHARLES POWELL WHITE (*J. Pathol. Bacteriol.*, 1908, 13, 3—11).—Crystals consisting of a loose combination of cholesterol with fatty acids, lecithin, or other substances occur in or among the cells of malignant tumours and in some other conditions. These crystals appear to be associated with cell proliferation rather than with degeneration. In degenerated areas, the crystals formed are mostly either cholesterol, fatty acids, or fats. It is suggested that cholesterol is associated with the regulations of cell proliferation.

W. D. H.

Peptonisation in Raw and Pasteurised Milk. RACHEL H. COLWELL and HENRY C. SHERMAN (*J. Biol. Chem.*, 1908, 5, 247—252).—The conclusion is drawn that pasteurisation is not an ideal process as a safeguard against infectious disease. Heating may destroy acid-producing organisms, but leaves intact putrefactive, ammonia-forming, and peptonising organisms. Pasteurisation at high temperatures (75—90°)

has less restraining effect on peptonisation and development of offensive odours than heating to 60°. The amount of ammonia does not always run parallel with intensity of the putrid odour. The determinations of intensity of odour, and also of peptone by the biuret reaction are admittedly rough, and, although conclusions are drawn in reference to infectious disease, no experiments or observations on pathogenic organisms appear to have been made.

W. H. D.

Action of Wines and of Alcohols on the Frog. VITTORIO NAZARI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 166—172).—In small doses, alcohols have no apparent physiological action on frogs, but in larger doses they produce narcosis or complete muscular relaxation, which lasts a longer or shorter time according to the concentration of the alcohol employed and to the size of the frog. Complete recovery takes place from this narcosis, but larger doses of the alcohols cause death. In the following list, the alcohols examined are arranged in the order of increasing narcotic power: ethyl, methyl, propyl, isobutyl, isoamyl, amyl. The alcoholic content of a wine can be determined roughly by ascertaining at what dilution it just causes or fails to cause narcosis, 1 c.c. of 4% ethyl alcohol having no narcotic influence even on small frogs; in this connexion, the presence in the wines of small proportions of highly narcotic higher alcohols must be borne in mind.

T. H. P.

The Mechanism of the Action of Arsenic Preparations on Trypanosomes in the Animal Organism. II. MARTIN JACOBY and ALBERT SCHÜTZE (*Biochem. Zeitsch.*, 1908, 13, 285—298).—Mice received injections of atoxyl, or of arsenious acid, and were then inoculated with *Nagana* trypanosomes. The trypanosomes from these animals were then inoculated into other animals similarly treated. This process of transference from animal to animal which had been injected with atoxyl or arsenious acid was repeated several times, and the sensitiveness of the trypanosomes obtained after many animals had been thus inoculated towards both arsenious acid and atoxyl was investigated, the experiments being carried out *in vitro*. No marked difference in this respect between these and normal trypanosomes could be ascertained.

S. B. S.

The Behaviour of Iron Arseno-paranuclate and of Arsenious Acid in the Organism. ERNST SALKOWSKI (*Biochem. Zeitsch.*, 1908, 13, 321—338).—The arseno-paranuclate was obtained by precipitating an arsenical peptic digestion product of caseinogen by ferric ammonium sulphate. There is evidence that the product obtained in this way is a definite compound, and not a mixture of ferric arsenate and paranuclate. Its behaviour in the organism was investigated by examining the urine after its administration, and determining the rate and the form of the arsenic excretion. It was found that if alcohol is added to alkaline urine, the arsenic in inorganic form is precipitated, whereas that in organic combination dissolves in alcohol. Iron arseno-paranuclate is readily resorbed from the digestive tract, a fact which confirms the assumption that the arseno-paranuclate is a definite

compound, for arsenate of iron is not readily resorbed. The arsenic excreted is chiefly in the form of an organic compound, although small quantities of inorganic arsenic can be detected in the urine in the first days after the administration. The arseno-paranucleate is also resorbed after subcutaneous injection. After administration of arsenious acid, both organic and inorganic arsenic is excreted, the former being in excess of the latter. Of ingested arsenious acid, at least 62% is excreted in the urine after six days. In some experiments, arsenic was detected in the urine of animals to which no arsenic compound had been directly administered; this is due to the small arsenic content of certain vegetables which served as food.

S. B. S.

Physiological Action of Choline. GEORG MODRAKOWSKI (*Pflüger's Archiv*, 1908, 124, 601—632).—This alkaloid appears to be less toxic than is usually considered to be the case. Many of the physiological effects, including the lowering of blood-pressure, usually attributed to it occur only in commercial specimens, but not in the pure substance. The action of the impurity is antagonised by atropine; hence it is that commercial choline produces, after atropinisation, a rise of blood-pressure, which is always the result if the pure alkaloid is employed. Pure specimens rapidly develop the impurity. The experiments were made with choline synthetically prepared.

W. D. H.

The Behaviour of the Brain towards Strychnine. TORATA SANO (*Pflüger's Archiv*, 1908, 124, 369—380).—The grey cortex of the human brain neutralises the toxic action of strychnine; this is especially marked in the motor areas, and the action is attributed in the main to the giant pyramid cells.

W. D. H.

Action of Strychnine and Caffeine. TORATA SANO (*Pflüger's Archiv*, 1908, 124, 381—391).—From experiments on frogs, the conclusion is drawn that strychnine has an anæsthetic as well as a stimulating action; the former manifests itself mainly on the pain receptive elements of the central nervous system, and the latter on the tactile elements; the stimulating action outlasts the anæsthetic, and is slower of development. Similar conclusions are drawn regarding action of caffeine.

W. D. H.

The Liver in Chloroform Necrosis (Delayed Chloroform Poisoning). GIDEON WELLS (*J. Biol. Chem.*, 1908, 5, 129—146).—Rapid autolysis of the liver cells, sometimes follows chloroform anæsthesia in man. A third or more of the solids may disappear in a few days, and the liver contains proteoses, purine bases, polypeptides, and amino-acids. The sulphur in insoluble form is unaltered, but, in spite of loss of nuclear structure, the insoluble phosphorus is increased. The distribution of nitrogen in mono- and di-amino-acids in the coagulated liver proteins does not differ from the normal. Fatty metamorphosis is present in moderate degree; the increase in ether extractives is due to infiltration with simple fats; the lecithin is

lightly decreased, and the amount of cholesterol unaltered. There is replacement of proteins by water, and more fatty infiltration than in acute yellow atrophy. W. D. H.

Chemistry of Vegetable Physiology and Agriculture.

The Chemical Changes Involved in the Assimilation of Free Nitrogen by *Azotobacter* and *Radiobacter*. JULIUS TOKLASA [with ADOLF ERNEST, FRANZ STRAÑAK and EUGEN VÍTEK] *Centr. Bakt. Par.*, 1908, 21, ii, 620. Compare this vol., ii, 880).—The activity of the culture decreases as it ages, doubtless because of the accumulation of acid. Quantitative experiments made to trace the fate of the dextrose were only partly successful; starting with 5.9 grams of dextrose, 7.9 were converted into carbon dioxide, 0.3 into ethyl alcohol, 0.2 into formic acid, 0.7 into acetic acid, 0.2 into lactic acid, leaving a balance of 6.5 unaccounted for, part of which, however, is assimilated into the cell wall of the organisms.

Contrary to the statements of Severin and Krzemieniewski, hydrogen is invariably liberated, and, if sodium nitrate has been added, a certain amount of nitrite and of ammonia is produced.

The author has made an analysis of the bacterial mass and found 11.3% and ash 8.6%; the latter contained P_2O_5 4.9, and K_2O 2.4, these two substances forming practically the whole of the ash.

E. J. R.

Fixation of Atmospheric Nitrogen by Pure Cultures of *Azotobacter*. Distribution of the Organism. MARTINUS J. BEYERINCK (*Proc. K. Akad. Wetensch. Amsterdam*, 1908, 11, 67—74. Compare Toklasa, this vol., ii, 880).—The author supposed previously that the active agent in nitrogen fixation is not *Azotobacter*, but *acillus radiobacter*, almost invariably found with it. His subsequent experiments have demonstrated the incorrectness of this view, and it is now clear that *Azotobacter* fixes nitrogen. The present paper deals with an improvement in technique, whereby it is possible to obtain a better idea than formerly of the distribution of the organism.

It is customary to use a sugar as the source of carbon in culture solutions, but the author finds that calcium malate is better; his solution was 100 tap water, 2 calcium malate, 0.05 K_2HPO_4 . Plates can be made if 1 to 2 parts of agar agar are also added; on these, a larger number of organisms develop into colonies than on any other plates, so that a more exact estimate of the number of *Azotobacter* present in a sample of soil can now be made. Instead of calcium malate, the lactate, acetate, or propionate can also be used, but in these media the organisms soon lose their power of growth, and inoculation to obtain pure cultures cannot be continued for long. Growth is also slow when calcium citrate, tartrate, or succinate is used, and there is no growth when calcium glycollate is supplied.

The amount of nitrogen fixed per gram of calcium salt oxidised was 4.9 mg. with the propionate, 2.8 mg. with the acetate, 2.6 mg. with the malate, and 1.8 mg. with the lactate. About 7 mg. are fixed for each gram of sugar oxidised when sugar is used as the source of carbon.

When calcium malate plate cultures are made of soil extracts, it is not usual to find colonies of *Azotobacter*, because only a small proportion survive and develop on plates, but when soil adhering to the roots of leguminous plants is examined, a number of colonies are produced, showing that a distinct relationship exists between the distribution of *Azotobacter* and the *Leguminosae*.

E. J. R.

Variation of the Rate of Disinfection with Change in the Concentration of the Disinfectant. HERBERT EDMESTON WATSON (*J. Hygiene*, 1908, 8, 536—542).—Miss Chick's results on the laws of disinfection, in which she showed that the disinfection is strictly analogous to a chemical reaction in which individual bacteria play the part of molecules, can be expressed by a mathematical formula, the use of which is illustrated by one of the problems worked out as follows: A solution of phenol containing 10 parts per 1000 disinfects a culture of *Bacillus paratyphosus* in twenty-five minutes; another solution takes thirty-five minutes. What is the strength of the second solution? Let the strength of the second solution be x ; for phenol, $n = 5.5$. Therefore $5.5 \log 10 + \log 25 = \text{constant} = 5.5 \log x + \log 35$. From this $x = 9.4$.

W. D. H.

Protein Formation in Ripening Seeds. N. WASSILIEF (*Ber. deut. bot. Ges.*, 1908, 26a, 454—467. Compare Abstr., 1901, ii, 185, and Nedokutschaeff, Abstr., 1902, ii, 281; 1903, ii, 508; Zaleski, 1905, ii, 549).—A continuation of work on the source and method of formation of protein in ripening seeds. As the result of a series of determinations, the amounts of nitrogen present in various forms, for example, as protein, asparagine, amino-acids, substances precipitated by phosphotungstic acid, &c., in (1) seeds at various stages of ripeness, (2) whole fruits, (3) husks of fruits, (4) whole leaves, (5) laminae, and (6) petioles, of *Lupinus albus*, the following principal conclusions are drawn.

After the removal of fruits from the plants, protein formation takes place in the former whether they are kept in the dark or in the light, and at the expense of asparagine, present in the fruit as plucked. The amino-acids at first increase slightly in amount, especially if the fruits are kept in the dark, doubtless as the result of protein decomposition, but eventually they are also used up, being probably transformed into asparagine, and then into protein. Organic bases play much the same rôle as amino-acids.

In the foregoing changes, the seeds become gradually richer in nitrogenous material, due to movement of nitrogenous substances from the husks of the fruits to the seeds. At the same time, the seeds become enriched with protein at the expense of the husks, so that the protein of the ripe seeds is, in part, derived from amino-compounds initially present in them when the fruits are plucked, and

in part, from amino-compounds subsequently formed in the husks. Seeds separated from the husks, and kept either dry or damp, form some protein from asparagine and other amino-compounds originally present in them.

T. A. H.

The Rapid Change in Composition of Certain Tropical Fruits during Ripening. H. C. PRINSEN GEERLIGS (*Proc. K. Akad. Wetensch. Amsterdam*, 1908, 11, 74—84).—The fruits investigated are commonly gathered in an immature state; within a few days they become tender and palatable; a few days later, however, they become over-ripe, and change to a soft, unpalatable mass. Experiments with bananas (*Musa*) showed that during the ripening process there is an evolution of carbon dioxide and water, and a considerable conversion of starch into sugar. The composition at the various stages is as follows:

	April 17, unripe.	April 19.	April 20.	April 22.	April 23, ripe.	April 24, over-ripe.
Dry matter, per cent...	41.76	40.79	40.52	40.14	39.02	38.88
Starch.....	30.98	24.98	20.52	13.80	9.59	7.68
Sucrose	0.86	4.43	6.53	10.50	13.68	10.36
Dextrose.....	0.25	0.96	1.80	3.18	4.72	6.1
Lævulose		0.90	1.53	2.70	3.61	4.8

Quite similar results were obtained with the mango (*Mangifera*) and the tamarind (*Tamarindus*).

Oxygen is necessary for the ripening process; bananas will keep their starch intact if surrounded with an atmosphere of nitrogen. The author, therefore, considers sugar production to be a vital process, although he succeeded in demonstrating the presence of an enzyme capable of decomposing starch.

Sapodilla (*Achras sapota*) does not fall into line with the above-mentioned fruits; the amount of sugar before and after ripening remains unaltered, and the change appears to be mainly a softening of the hard pectin, and a deposition of tannin and gutta-percha from the juice as insoluble substances.

E. J. R.

Vegetable Phosphatides. ERNST SCHULZE (*Chem. Zeit.*, 1908, 32, 981—983. Compare this vol., i, 385).—Phosphatides were determined in a number of seeds (without husks), and the following results obtained (percentages in dry matter):

	Phosphorus.		Phosphatides as lecithin.
	Total per cent.	Per cent. in ether-alcohol.	
Yellow lupins	—	0.082	2.14
Blue „	1.53	0.084	2.19
Garden beans	1.32	0.049	1.27
<i>Phaseolus multiflora</i>	—	0.035	0.90
Sunflower	—	0.017	0.44
<i>Pepo cucurbita</i>	2.10	0.021	0.55
<i>Ricinus</i>	1.14	0.011	0.29
Beech	—	0.011	0.30
Chestnut	—	0.026	0.67
Horse chestnut	—	0.026	0.67
<i>Pinus cembra</i>	1.16	0.038	0.99
<i>Pinus maritima</i>	2.60	0.033	0.86

The phosphatide represents only a small percentage of the total phosphorus (in the case of *Ricinus*, only 1%), and, since the seeds contain very little inorganic phosphorus, it is evident that some other organic phosphorus compounds must be present in much larger quantity than the phosphatides. The substance known as phytin, which yields inositol as a cleavage product, is one of these substances, and is presumably of greater importance to plants than the phosphatides.

N. H. J. M.

Water-soluble Polysaccharides of Barley and Malt. HORACE T. BROWN (*Bied. Zentr.*, 1908, 37, 675—676; from *Zeitsch. ges. Brauwesen*, 1907, 30, 286).—Finely-crushed barley (1 kilo.) was stirred with boiling water (7 litres), cooled to 70°, and, after adding 15 c.c. of malt extract, boiled for a long time. A further amount (250 c.c.) of malt extract was then added, and the whole digested for one hour at 50—55° and again boiled. It was then filtered, the residue well washed with hot water, and the filtrate evaporated to 4 litres under reduced pressure. The resulting liquid, D. 1.060, containing 15% of dry matter, was treated with 80% alcohol; on cooling, white flakes of crude amylan separated. The latter, after being thoroughly washed with 60—70% alcohol, was treated with 500 c.c. of water, and again precipitated with alcohol.

The soluble constituents of barley are as follows: ash, 0.75; proteins, 0.75; sugar, 4.10; starch, 56.20, and amylan, 9.65%. The amylan yields, when hydrolysed with 2.5% oxalic acid, chiefly dextrose along with galactose, mannose, arabinose, and xylose.

Crude amylan from malt dissolves readily in cold water, is strongly dextrorotatory, and has slightly reducing properties. When hydrolysed, it yields dextrose and arabinose.

N. H. J. M.

Fruits of *Caulophyllum thalictroides* and *Cornus sericea*. EDITH STOCKTON and C. G. ELDREDGE (*Chem. News*, 1908, 98, 190—191).—The pulp of the fruit of *Caulophyllum thalictroides* contains levulose and small quantities of citric, tartaric, and tannic acids. The crushed nuts yield to ether about 3% of oil, calculated on the whole fruits, of which a portion is volatile and the remainder belongs either to the laurin or olein group.

The pulp of the fruit of *Cornus sericea* contains potassium hydrogen tartrate, potassium hydrogen oxalate, calcium oxalate, gum, tannic, gallic, and malic acids, and a sugar, possibly levulose. The nuts yield to ether a pale amber-coloured oil, which deposits some solid matter, perhaps palmitin, on standing.

T. A. H.

Constituents of the Rhizome of *Imperatoria ostruthium*. JOHANNES HERZOG (*Arch. Pharm.*, 1908, 246, 414—417).—The author has applied the method devised for the preparation of pimpinellin (this vol., i, 905), namely, extraction with benzene and treatment of the concentrated extract with light petroleum, to this rhizome, and has isolated thereby oxypeucedanin, identical with that prepared by Erdmann and by Bothe from the root of *Peucedanum*

officinale. Gorup-Besanez (Abstr., 1874, 907; 1877, 717) obtained ostruthin as the principal constituent of the young rhizome of *Imperatoria ostruthium*, although Heut subsequently (this Journ., 1875, 772) detected oxypeucedanin in the rhizome. It remains to be seen whether ostruthin and oxypeucedanin both occur always in the rhizome, or whether the former is replaced by the latter as the rhizome ages or is collected and stored.

T. A. H.

Constituents of Saffron. BALTHASAR PFYL and W. SCHEITZ (*Zeitsch. Nahr. Genussm.*, 1908, 16, 337—346. Compare Hilger, Abstr., 1900, i, 682).—With the object of devising a process for the detection of inferior saffron (this vol., ii, 997), attempts were made, following lines suggested by the results of previous investigators, to obtain data regarding the properties of the chief constituents of the drug.

The methods suggested by Quadrat (*J. pr. Chem.*, 1852, 56, 68) and Weiss (*ibid.*, 1867, 101, 65), involving the extraction of the saffron, previously freed from fat, &c., by means of ether or light petroleum, with water, and precipitation of the colouring matter (polychroit or crocin) from the aqueous extract by lead acetate or alcohol, do not yield a pure preparation, and the same is true of Kayser's process, depending on the use of animal charcoal for the extraction of the colouring matter (Abstr., 1885, 59). Only amorphous, impure preparations of crocin could be obtained, and these, on hydrolysis with acids, furnished dextrose (compare Kayser, *loc. cit.*; Schunck and Marchlewski, Abstr., 1894, i, 340).

Similarly, Kayser's crocetin (*loc. cit.*) could only be obtained as an amorphous, red mass, which, however, yielded crystalline salts with metals and certain organic bases (compare Decker, Abstr., 1906, i, 686).

Kayser's picrocrocin (*loc. cit.*) could not be prepared, although by using his process for its isolation a small amount of a white, crystalline substance, m. p. 67°, which did not reduce Fehling's solution, was obtained.

From a chloroformic extract of saffron, three substances were isolated: (1) a colourless, crystalline product, m. p. 280°, which did not reduce Fehling's solution; (2) a yellow, crystalline substance, m. p. 164°, readily soluble in water or alcohol, which when boiled with acid developed the odour of saffron oil and yielded a reducing sugar, probably lævulose, and (3) a crystalline hydrocarbon, m. p. 118° (compare Schüler, *Inaug. Diss. Munich*, 1899, and Hilger, *loc. cit.*), apparently similar to the hydrocarbons found in marigold petals, amica flowers, &c. The second of these products somewhat resembles the picrocrocin described by Kayser.

An alcoholic extract of saffron contains, in addition to colouring matter, a sugar (? lævulose) and a glucoside, which, on hydrolysis yields saffron oil and lævulose (?). The sugar provisionally regarded as lævulose is levorotatory, reduces Fehling's solution, yields phenylglucosazone, and gives the Seliwanoff reaction (Abstr., 1903, ii, 616).

T. A. H.

Have Manganese Salts, Employed as Stimulants, a Favourable Influence on Vegetation? SIGURD RHODIN (*Bied. Zentr.*, 1908, 37, 667—668; from *K. Landtbr. Akad. Handl. Tidskr. Stockholm*, 1908, 30—33).—Field experiments with oats grown on peaty soil and with potatoes on rich garden soil. Manganese peroxide (1 kilo. per are), manganese acetate, and benzoate (30 grams per 100 square metres) reduced the yield of oats. In the potato experiment, manganese sulphate (6 kilos. per hectare) was employed. One variety of potatoes showed an increase of 1% of starch on the manganese plot; the total starch was, however, considerably less than without manganese, owing to the smaller yield of tubers. N. H. J. M.

Manurial Experiments with Nitrogen in 1907. HENRIK G. SÖDERBAUM (*Bied. Zentr.*, 1908, 37, 657—659; from *K. Landtbr. Akad. Handl. Tidskr. Stockholm*, 1908, 104—110).—Oats were grown in sandy soil (25 kilos. per pot), and manured with 0.25, 0.5, and 0.75 gram of nitrogen in different forms, in addition to minerals. The relative manurial effects of the nitrogen compounds varied according to the amounts employed. With the smallest amounts, sodium nitrate gave the highest yields. When 0.5 gram of nitrogen was applied, ammonium sulphate gave the best results; then Polzeniusz's, Carlson's, and Frank's calcium cyanamide preparations; the next best were sodium nitrate and albumin (about equal), and last, calcium nitrate. With the largest amounts of nitrogen, ammonium sulphate was again the best; then Carlson's and Polzeniusz's calcium cyanamide; Frank's cyanamide gave considerably less produce, whilst calcium nitrate was again the least satisfactory.

The total divergence of the results from those of the previous year is attributed to the unusually low temperature in 1907, and the consequent more prolonged vegetative period. N. H. J. M.

Field Experiments with Ammonium Sulphate. HERMANN BACHMANN (*Bied. Zentr.*, 1908, 37, 664—665; from *Fühling's Landw. Zeit.*, 1907, 530).—Experiments with rye, oats, barley, and mangolds grown on sandy soil and on loam showed that ammonium sulphate produced considerably greater yields than sodium nitrate. N. H. J. M.

Utilisation of Nitrogen in the Form of Ammonium Nitrate. THEODOR PREIFFER, A. HEPNER, and L. FRANK (*Bied. Zentr.*, 1908, 37, 663—664; from *Mitt. Landw. Inst. K. Univ. Breslau*, 1908, 5, 341).—The plants were grown in sand, and manured with ammonium nitrate and sulphate and sodium nitrate respectively. The position of ammonium nitrate as a manure is between sodium nitrate and ammonium sulphate; it may, however, under certain conditions, equal sodium nitrate. Addition of sodium chloride had no appreciable effect. The favourable results obtained by Wagner with sodium chloride in conjunction with ammonium sulphate may have been due to the presence of zeolites in the soil.

Addition of phosphorite along with ammonium nitrate increased the yield and also the amount of nitrogen taken up. N. H. J. M.

Manurial Trials with Sodium Nitrate and Ammonium Sulphate. H. CLAUSEN (*Bied. Zentr.*, 1908, 37, 585; from *Illustr. landw. Zeit.*, 1907, 27, 842).—Ammonium sulphate proved more effective than sodium nitrate on potatoes, rye, and oats growing on sandy soils. The superiority may be accounted for in several ways; there are indications that the potato plant can directly assimilate ammonia, so that nitrification becomes unnecessary; it is also known that sodium nitrate washes into the sub-soil, and is therefore lost more readily than ammonium sulphate. On the soils in question, Wagner's generalisation, that ammonium sulphate is only 75% as effective as sodium nitrate, clearly does not hold, and the author is of opinion that results similar to his own would commonly be obtained elsewhere.

E. J. R.

Plot Experiments on the New Nitrogenous Manures. HJALMAR VON FEILITZEN (*Bied. Zentr.*, 1908, 37, 659—663; from *Svenska Mosskulturförening. Tidskr.*, 1908, 91—108).—Calcium nitrate produced higher yields of potatoes and of starch than potassium nitrate both on incompletely humified *Sphagnum* soil deficient in nitrogen, and on peat soil rich in nitrogen. Similar experiments with oats grown in sandy peat gave similar results.

Experiments in which different varieties of potatoes were grown in sandy soil, and manured respectively with sodium nitrate and two calcium cyanamide preparations, showed that, whilst the plants manured with cyanamide developed somewhat better than those on the nitrate plots, the final yields both of potatoes and starch were highest where nitrate had been supplied.

N. H. J. M.

Analytical Chemistry.

A Shortened Burette. F. TSCHAPLOWITZ (*Zeitsch. anal. Chem.*, 1908, 47, 697—698).—The apparatus consists of two 25 c.c. burettes connected at their lower end with a single tap. When the contents of one of the burettes has been run off through the tap, a half-turn of the latter enables the contents of the second burette to be used.

W. P. S.

Combined Wash-bottle and Pipette. J. W. HOGARTH (*J. Roy. Soc., New South Wales*, 1905, 38, 418—420).—A modified wash-bottle by means of which it is possible to deliver an exactly measured volume of liquid.

P. H.

Destruction of Organic Substances. M. KERBOSCH (*Pharm. Weekblad*, 1908, 45, 1210—1213).—The author emphasises the importance of the absence of organic matter in testing for metallic

poisons, and describes a method for destroying it by heating the material with a mixture of sulphuric acid and nitric acid. The method has been applied to milk, peas, meat, and sardines.

A. J. W.

The Use of Nitrous Acid, Nitrites, and Aqua Regia in the Estimation of the Mineral Constituents of Urine. JOSEPH H. KASTLE (*Amer. J. Physiol.*, 1908, 22, 411—422).—The difficulty of incinerating urine completely is well known. If, however, the urea is first got rid of by heating with sodium nitrite, nitrous acid, or aqua regia, incineration is rapidly accomplished, and the inorganic constituents of the ash can be estimated accurately. Each of the three reagents mentioned has special advantages in certain cases.

W. D. H.

Action of Thiosulphate on Permanganate in Alkaline Solution. HEINRICH KILIANI (*Chem. Zeit.*, 1908, 32, 1018).—Reinige proposed to estimate alkali iodides by titration with permanganate (conversion into iodate), and to estimate the excess by means of thiosulphate, when tetrathionate was supposed to be formed. The author, having received a private communication from Herms, has had the matter investigated, and now states that in the oxidation of thiosulphate in alkaline solution, sulphate is formed. Hence, when using Reinige's process, it must be remembered that 8 mols. of potassium permanganate do not require 24, but only 3, mols. of sodium thiosulphate for decolorisation.

L. DE K.

Estimation of [Organic] Sulphur by Carius' Method. ERWIN RUPP (*Chem. Zeit.*, 1908, 32, 984).—It is recommended to add barium chloride when oxidising the substance with nitric acid instead of adding it when the oxidation is finished. The barium sulphate thus obtained is of a coarse structure, and consequently readily washed and collected. It must be remembered that it always contains barium nitrate, from which it may be freed by boiling with a 150—200 c.c. of water, any large particles being broken up with a glass rod.

L. DE K.

Volumetric Estimation of Sulphuric Acid. THOMAS COCKSEY (*J. Roy. Soc., New South Wales*, 1907, 41, 215—217).—The author has improved the titration of barium and calcium salts by means of $N/10$ sodium carbonate and phenolphthalein as indicator, by adding alcohol in such quantity that, after the experiment is finished, the liquid will contain about half its bulk of it. Before titrating, the liquid must, if necessary, be rendered neutral to phenolphthalein with potassium hydroxide. The barium or calcium carbonate is immediately precipitated, and the end reaction becomes very distinct.

Sulphates (if necessary, freed from metals other than alkalis) are estimated conveniently by adding to the carefully-neutralised solution a definite amount of barium chloride, the excess of which is then estimated by the above process.

L. DE K.

Modification of Hüfner's Method for the Volumetric Estimation of Nitrogen. VICTOR VON CORDIER (*Zeitsch. anal. Chem.*, 1908, 47, 682—687).—The apparatus used consists of a cylindrical bulb with a long neck, which is provided with a side-tube, the whole being similar to a Victor Meyer's vapour density apparatus. The bulb has a capacity of about 100 c.c., and is fitted with a stoppered tubulure for introducing the sodium hypobromite solution. A second bulb is also provided, and serves as a reservoir for a further quantity of the solution; it is attached to the main bulb by a tapped tube, which enters the shoulder of the main bulb, and also at its top by a second tapped tube, which is connected with the neck of the apparatus. About 50 c.c. of Knop's (sodium hypobromite) solution are placed in the main bulb, and about 40 c.c. in the reserve bulb. When required, this portion of the solution is allowed to enter the main bulb by opening both the taps. The use of the apparatus enables a larger quantity of substance to be taken for the estimation, and results of experiments are given showing that the method may be used for the estimation of nitrogen in guanidine picrate, urea nitrate, acetylurea, ammonium platinichloride, &c. W. P. S.

Estimation of Ammonia in Urine. A. RONCHÈSE (*Bull. Soc. chim.*, 1908, [iv], 3, 840. Compare *Abstr.*, 1907, ii, 651).—A claim for priority against Malfatti (this vol., ii, 531). E. H.

Presence of Nitrite and Ammonia in Well-water and Its Signification. J. VAN EYK (*Pharm. Weekblad*, 1908, 45, 1162—1165).—At the ordinary temperature, zinc reduces nitrates in aqueous solution to nitrites, and then to ammonia. The author considers that nitrates in well-water may be reduced to nitrites by the zinc employed to galvanise the iron pipes of water pumps. A. J. W.

Estimation of Phosphorus, Sulphur, and Silicon in Acetylene. ADOLF FRAENCKEL (*Chem. Zentr.*, 1908, ii, 643—644; from *J. Gasbeleuchtung*, 1908, 51, 431—435).—The acetylene generated from a known weight of calcium carbide is burnt in a special apparatus, and the products of combustion are drawn through sodium hypobromite solution.

On evaporating to dryness with addition of hydrochloric acid, the silica is obtained; in the filtrate from this, the phosphoric acid is estimated as usual with magnesia mixture. Sulphur is estimated similarly by passing the products of combustion through sodium hypobromite and estimating the sulphuric acid formed as usual.

L. DE K.

Estimation of Phosphoric Acid by Lorenz's Method. OTTOKAR FALLADA (*Chem. Zentr.*, 1908, ii, 827—828; from *Osterr.-ung. Zeitsch. Zucker-Ind. Landw.*, 1908, 37, 333—336).—Lorenz's method (direct weighing of the yellow precipitate) is recommended, and will be found useful for the estimation of citrate-soluble phosphoric acid.

L. DE K.

Estimation of Arsenic in Iron Ores. MARCEL GUEDRAS (*Chem. Zentr.*, 1908, ii, 444; from *Rev. gener. Chim. pure appl.*, 1908, 11, 251—253).—One gram of the finely-powdered ore is boiled in a 500 c.c. flask with 150 c.c. of hydrochloric acid and 5 grams of stannous chloride, and the distillate is collected in a graduated 100 c.c. receiver containing 50 c.c. of water.

When 40 c.c. have passed over, the distillate is nearly neutralised, a few grams of sodium hydrogen carbonate are added, and the arsenic is titrated as usual with standard iodine.

L. DE K.

Apparatus for the Estimation of Carbon in Iron. MAX WIDEMANN (*Chem. Zentr.*, 1908, ii, 724—725; from *Zeitsch. Chem. Apparatenkunde*, 1908, 3, 296).—An improved flask and condensation arrangement. The essential improvement consists in the condensing tube being fitted with 5 bulbs. The gaseous products escape through a side-tube. The tube carrying off the water is fitted with a funnel, in which the water rises in case of sudden pressure, thus preventing the tube from cracking.

L. DE K.

Simultaneous Estimation of Carbon, Hydrogen, and Nitrogen, &c., in Organic Compounds by the Method of Simplified Elementary Analysis. MAX DENNSTEDT and F. HASSLER (*Ber.*, 1908, 41, 2778—2782).—Oxygen, prepared by heating potassium permanganate, passes through a sulphuric acid tube, through one limb of a T-piece, and through a soda-lime-calcium chloride tube into the bifurcated entry-tube of the combustion apparatus. Through the other limb of the T-piece, oxygen is driven into a weighted rubber bag (15 kilo. per 300 sq. cm.) which acts as a reservoir, by which the current of oxygen during the combustion can be adjusted to a nicety. The combustion tube and absorption apparatus are arranged as usual. To absorb oxygen, a 1 litre Erlenmeyer flask is provided with a two-holed stopper, through which one tube, passing to the bottom of the flask, is connected with an ordinary pressure-adjusting bulb, whilst a capillary T-piece, passing just through the other hole, is connected by one limb with the combustion apparatus, a safety bottle being inserted to prevent back-suction; the other limb of the T-piece, provided with rubber tubing and a pinch-cock, serves as a gas-exit.

The best absorbent of oxygen is cuprous chloride in hydrochloric acid containing pieces of copper gauze. The solution is prepared best from copper sulphate and an excess of hydrochloric acid, as the presence of sulphuric acid increases the rate of absorption of the oxygen. The solution is efficient so long as copper gauze remains undissolved.

The expulsion of air from the whole apparatus requires one to one and a-half hours. At the completion of the combustion the nitrogen is swept into the Erlenmeyer flask for about twenty minutes; after standing overnight, the nitrogen is measured in a Hempel burette. Any nitrogen retained as lead nitrate by the lead peroxide must be extracted by 33% alcohol, the solution evaporated, and the residue of lead nitrate weighed. Sulphur and the halogens are estimated as described in previous communications.

The method is most suitable for the analysis of costly substances, difficult to obtain pure. C. S.

Volumetric Estimation of Sodium Hydroxide in the Presence of Sodium Carbonate. A. C. ANDERSEN (*J. Pharm. Chim.*, 1908, 28, 370—371; from *Tidskr. Kem. Farm. Terapi*, 1908, 11, 161).—This estimation is generally carried out by adding barium chloride, which precipitates the carbonate and leaves the hydroxide; the latter is then titrated with standard acid and phenolphthalein as indicator. But in order to obtain trustworthy results, the author proposes the following slight modification.

After operating in the manner described, another portion of the solution is taken, and sufficient standard acid is added to neutralise the greater part of the hydroxide. The liquid is then heated to boiling, and a slight excess of barium chloride solution is added. When the liquid has cooled (the flask being closed), the titration is continued with phenolphthalein as indicator. L. DE K.

Ready Means of Comparing Sodium Carbonate and Oxalic Acid Solutions. A. TIAN (*Chem. Zentr.*, 1908, ii, 636; from *Rev. gen. Chim. pure appl.*, 1908, 11, 208).—The process is based on the fact that oxalic acid liberates the mineral acid from a calcium salt. A measured amount of the oxalic acid solution to be tested is mixed with an excess of calcium chloride, and the turbid liquid titrated with sodium carbonate solution, using helianthin as indicator.

L. DE K.

Estimation of Small Amounts of Barium in Rocks. RALPH W. LANGLEY (*Amer. J. Sci.*, 1908, [iv], 26, 123—124).—The author estimates small amounts of barium in rocks by precipitation with sulphuric acid immediately after the separation of silica. It is necessary to re-dissolve the barium sulphate in concentrated sulphuric acid, and to precipitate it with water, in order to free it from ferric and other sulphates. If barium is not removed as sulphate, it is precipitated as phosphate, and introduces an error in the estimation of magnesium. R. J. C.

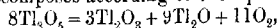
Separation of the Metals which are Precipitated by Hydrogen Sulphide. H. BOLLENBACH (*Zeitsch. anal. Chem.*, 1908, 47, 690—693).—The metals which are precipitated by hydrogen sulphide, and are insoluble in ammonium sulphide, namely, mercury, lead, bismuth, cadmium, and copper, may be separated from each other by the following method. The precipitated sulphides are treated with nitric acid, when all but the mercury goes into solution. Ammonium persulphate is added to the solution, then a considerable excess of ammonia, and the mixture is boiled. Lead and bismuth are precipitated and removed by filtration. The precipitate is dissolved in concentrated hydrochloric acid; one portion of the solution is rendered ammoniacal, then acidified with acetic acid, and tested with potassium dichromate, a yellow precipitate denoting the presence of lead (bismuth chromate is readily soluble in acetic acid).

A second portion of the hydrochloric acid solution is treated with stannous chloride, and sufficient sodium hydroxide is added to redissolve the stannous hydroxide. A black precipitate shows that bismuth is present. The filtrate from the lead and bismuth peroxides is boiled with hydrochloric acid to decompose the ammonium persulphate, and then rendered ammoniacal. A blue coloration denotes the presence of copper. The blue solution is then treated with potassium cyanide and hydrogen sulphide, when a yellow precipitate is obtained if cadmium is present.

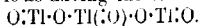
W. P. S.

Electrolytic Estimation of Thallium and Probable Existence of a New Oxide of this Metal. GINO GALLO and G. CENNI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 276—284).—When a solution of thallous sulphate, slightly acidified with sulphuric acid, is electrolysed, the whole of the thallium is deposited on the anode in the form of an oxide, generally supposed to be the sesquioxide. It was found, however, by Heiberg (*Abstr.*, 1903, ii, 614) that, when the deposit of oxide is heated in order to dry it, its weight at first diminishes and subsequently increases, this increase being supposed by him to be due mainly to the action on the oxide of sulphur dioxide from the gas heating the oven, whilst Werther (*J. pr. Chem.*, 1864, 91, 385) regarded it as being caused by the absorption of carbon dioxide. The authors find that the minimum weight of the deposit is greater than that of the sesquioxide corresponding with the amount of thallium employed, their results indicating that the sesquioxide is converted into an oxide of the composition Tl_3O_5 . The oxide Tl_3O_5 is probably transformed partly into TlO_2 , which, with Tl_2O_3 , gives the compound Tl_3O_5 . The formation of such an oxide is not surprising, considering the position of thallium between mercury and lead in the periodic system, and also the existence of mercury peroxide (compare Antropoff, *Zeitsch. Elektrochem.*, 1906, 12, 585; Pellini, *Gazzetta*, 1908, 38, i, 71).

The best procedure for the quantitative, anodic deposition of thallium as oxide is to dissolve thallous sulphate in about 100 c.c. of water in a Classen capsule, the solution being acidified by the addition of about 0.1 gram of oxalic acid, and electrolysed at the ordinary temperature with a platinum disk rotating at 800 revs. per minute as negative electrode; the voltage should be 3—4, and the current density, 0.15—0.20 ampere. The end of the electrolysis is ascertained by adding a little water so as to raise the level of the liquid in the capsule; no deposit should appear on the clean platinum surface thus freshly brought into contact with the solution. The weight of the deposit, after drying at 160—200°, corresponds with the formula Tl_3O_5 , and, when dissolved in hydrochloric acid, the oxide causes evolution of chlorine and forms the chloride, $TlCl_3 \cdot 3TlCl$. The oxide hence decomposes according to the equation:



and the authors regard it as having the structure



T. H. P.

Analysis of a Mineral containing Copper, Bismuth, Lead, Silver, Calcium, Iron, and Quartz. AUGUST CHWALA and V. MACRI (*Chem. Zentr.* 1908, ii, 261; from *Mon. Sci.*, 1908, [iv], 22, 372).—The mineral is decomposed by means of nitric acid and potassium chlorate, and a solution of ammonium nitrate is added to keep lead and calcium sulphates in solution. The siliceous matter is then collected and washed, first with a hot acid and then with a hot ammoniacal solution of ammonium nitrate. After removing any silver from the filtrate by means of hydrochloric acid, sulphuric acid is added, and then excess of ammonia, which precipitates the iron only; this is washed first with an acid and then with an ammoniacal solution of ammonium nitrate. The bismuth is then precipitated with ammonium carbonate, and, finally, the copper and lead are separated as usual.

L. DE K.

Solubility of Rare Earth Oxalates in Solutions containing Uranyl Salts. OTTO HAUSER (*Zeitsch. anal. Chem.*, 1908, 47, 677—680).—It is pointed out that uranyl salts have a considerable solvent action on cerium and lanthanum oxalates, and, unless a considerable excess of oxalic acid is added, the results obtained in the usual method of precipitating these earths as oxalates are untrustworthy, if uranyl salts are present.

W. P. S.

Separation of the Metals of the Ammonium Sulphide Group. ERICH EBELER (*Zeitsch. anal. Chem.*, 1908, 47, 665—677).—The following process of separating the metals precipitated by ammonium sulphide is recommended. The precipitate, consisting of iron, manganese, nickel, cobalt, and zinc sulphides, and aluminium and chromium hydroxides, is boiled with hydrochloric acid until all the hydrogen sulphide has been expelled, then oxidised with nitric acid, and treated with hydrogen peroxide and sodium hydroxide. The precipitate, which contains the iron, manganese, nickel, and cobalt, is separated by filtration, dissolved in hydrochloric acid, hydrogen peroxide is added, and the iron and manganese are precipitated with ammonia; these two metals are separated from their hydrochloric acid solution by the addition of ammonium chloride, hydroxylamine hydrochloride, and ammonia, the iron being precipitated, whilst the manganese remains in solution. The nickel and cobalt are separated by adding dicyanodiamide and potassium hydroxide to the filtrate containing them; the nickel is precipitated as a crystalline compound. The filtrate containing the aluminium, chromium, and zinc is boiled, and ammonium chloride is added repeatedly until ammonia ceases to be given off; the aluminium hydroxide thus precipitated is removed by filtration, and the filtrate is treated with sulphurous acid after acidifying with hydrochloric acid. The addition of ammonia to the hot solution precipitates the chromium, leaving the zinc in solution.

W. P. S.

Estimation of Iron, Alumina, and Phosphoric Acid in Presence of Each Other. THOMAS COCKSEY (*J. Roy. Soc., New South Wales*, 1907, 41, 163—171).—The acid solution is mixed with a definite

amount of a standard solution of sodium dihydrogen phosphate, and potassium hydroxide solution is added until the liquid is neutral to methyl-orange. The whole is diluted to 80—90 c.c., and heated on the water-bath until the precipitate has settled. To the filtrate is added an excess of calcium chloride, the liquid is acidified slightly with $N/10$ acid, boiled to expel carbon dioxide, and the free phosphoric acid is titrated with $N/10$ potassium hydroxide, using phenolphthalein as indicator, as soon as the liquid is neutral to methyl-orange; 1 c.c. = 0.00355 gram of P_2O_5 .

The iron oxide is determined iodometrically, and calculated to ferric phosphate. This deducted from the weight of the first precipitate gives the aluminium phosphate, which is then calculated to alumina. From the total phosphoric acid found, is then deducted the amount purposely added.

L. DE K.

Separation of Iron from Nickel and Cobalt by Lead Oxide. T. H. LABY (*J. Roy. Soc., New South Wales*, 1904, 37, 157).—After trying the separation by means of ammonium hydroxide and chloride, ammonium carbonate, the basic acetate process, the phosphate method, the electrolytical separation, and the ether extraction process, the author finally calls attention to a process given by Field (*Chem. News*, 1859, 1, 5), which he finds is also suitable for the separation of iron from cobalt.

In this process the iron is precipitated as hydroxide by evaporating the nitrate solution with excess of lead oxide; the lead is separated readily from the filtrate with sulphuric acid. The nickel or cobalt is then estimated electrolytically in ammoniacal solution.

L. DE K.

Detection of Cobalt in the Presence of Large Quantities of Nickel. M. EMMANUEL POZZI-ESCOT (*Ann. Chim. anal.*, 1908, 13, 390—391).—Supposing the amount of nickel to be about 1000 times that of the cobalt, the solution containing about 10 grams of the nitrates is concentrated to 25—50 c.c. and mixed while hot with 50 grams of ammonium molybdate dissolved in a little boiling water. When cold, the liquid is filtered from the nickel precipitate, and the rose colour of the cobalt becomes visible. On boiling with excess of sodium hydroxide and a little hydrogen peroxide, the cobalt is obtained as a brown oxide, which may then be identified by means of the borax bead.

L. DE K.

Approximate Colorimetric Estimation of Cobalt and Nickel in the Presence of Each Other. R. W. CHALLINOR (*J. Roy. Soc., New South Wales*, 1905, 38, 406—417).—The two metals are estimated jointly by electrolysis, and then dissolved in dilute nitric acid (1:1). After evaporating nearly to dryness, the mass is dissolved in water and diluted to a known volume. If the liquid is green, more than 76% of nickel is present; an aliquot portion of the liquid, containing 0.04—0.05 gram of the mixed metals, is taken, and a standard solution of cobalt is run in until the colour (after diluting to the 50 c.c. mark) is equal to that of a standard solution containing the nearest weight of the mixed metals. If, however, the solution is pink, showing

the presence of more than 24% of cobalt, standard nickel is run in, any excess of which may be again checked with cobalt.

After allowing for nickel added, the amount of nickel in the mixture is readily calculated.

L. DE K.

Iodometric Estimation of Chromic and Vanadic Acids in the Presence of Each Other. GRAHAM EDGAR (*Amer. J. Sci.*, 1908, [iv], 26, 333—336).—The mixed acids (about 0.2 gram) are boiled with 15 c.c. of hydrochloric acid and 2 grams of potassium bromide in a Voit flask, a slow current of hydrogen is passed, and the bromine evolved, owing to the reduction of 2CrO_4 to Cr_2O_3 and V_2O_5 to V_2O_4 , is absorbed in an alkaline solution of potassium iodide contained in a Drexel bottle connected with a Will and Varrentrap trap, also containing the same solution. After the operation is over, the potassium iodide solution is acidified with hydrochloric acid, and the iodine set free titrated as usual. The Drexel bottle is then filled with fresh potassium iodide solution.

To the contents of the Voit flask are now added 2 grams of potassium iodide, 15 c.c. of hydrochloric acid, and 3 c.c. of syrupy phosphoric acid, and the distillation is repeated as before in a current of hydrogen. The hydriodic acid acts on the V_2O_4 , reducing this to V_2O_3 with liberation of iodine, which is absorbed in the Drexel bottle and titrated as usual. The second titration therefore gives the vanadic acid only, and the difference between the first and second gives the necessary data for the calculation of the chromic acid.

L. DE K.

Tin. DAVID B. DOTT (*Pharm. J.*, 1908, [iv], 27, 486).—Contrary to general belief, metastannic acid, formed by the action of nitric acid on tin, is soluble in hydrochloric acid. All quantitative processes based on the separation of silica from tin by evaporation to dryness are untrustworthy, as a large proportion of the tin volatilises as chloride. The author proposes the following method for the estimation of tin in the presence of antimony.

The solution is divided into two equal portions. In one of these, both metals are precipitated with hydrogen sulphide, and the precipitate is then converted in the usual manner into a mixture of tin and antimony dioxides. The other portion is saturated with oxalic acid, heated, and treated with hydrogen sulphide, which now precipitates the antimony only; this is then converted into dioxide and deducted from the joint weight.

Low's modification of Pearce's method for the estimation of tin in ores, by fusing with sodium hydroxide, dissolving in hydrochloric acid, reducing by boiling with iron wire, and, after filtering into a flask filled with carbon dioxide, rapidly titrating with standard iodine, gives good results.

L. DE K.

Analysis of Oxidised Antimony and Lead Sulphide Compounds. FELIX JACOBSON (*Chem. Zeit.*, 1908, 32, 984—985).—The article is mainly devoted to the assay of commercial Sulphur antimonii auratum (this vol., ii, 540).

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The process is also applicable to lead sulphide. For the estimation of free sulphur, the use of boiling chloroform is recommended.

L. DE K.

Volumetric Estimation of Bismuth. PIERRE BALAVOINE (*Zeitsch. anal. Chem.*, 1908, 47, 681).—The author mentions that previous to the publication of the results of Moser's investigation (*Abstr.*, 1907, ii, 433) on the volumetric estimation of bismuth, he had recorded the results of experiments showing that the method was untrustworthy.

W. P. S.

Analysis of Organic Mixtures with the Aid of the Refractometer. ERNST E. SUNDWICK (*Pharm. Zentr.-h.*, 1908, 49, 783—787).—Some remarks on the process of Beythien and Hennicke (this vol., ii, 72).

The author's own process is based on the following equations: (1) $V:V_1 = C_1 - B : C - B$, in which V and V_1 represent two volumes containing the same amount of dissolved matter, C the refraction of the joint solution, and B the refraction of the solvent (water, alcohol, chloroform, acetone, toluene, &c.); (2) $V(C - B) = \text{constant}$, and (3) $P(C - B)/d = \text{constant}$.

L. DE K.

Methyl Alcohol and its Impurities. FRANZ FRIEDRICH (*Chem. Zeit.*, 1908, 32, 890—891).—In examining methyl alcohol for impurities, the carbylamine test for chloroform should not be omitted, in view of the fact that methyl alcohol is sometimes treated with bleaching powder in order to remove acetone, which is thereby converted into chloroform.

P. H.

Gravimetric Alcoholometry. BLONDEAU (*Bull. Assoc. Chim. Sucr. Dist.*, 1908, 26, 148—160).—Tables are given showing the weight of alcohol per hectolitre of mixtures of alcohol and water. The values are given for each reading of the Gay-Lussac alcoholometer between 1 and 100 at temperatures from 0° to 30° (compare *Abstr.*, 1908, ii, 738).

W. P. S.

New Differential Reactions of the Naphthols. VOLER-BOUCHER (*Ann. Chim. anal.*, 1908, 13, 335—338).—The following reagents are used: Alcohol of 95° (French), a 10% solution of copper sulphate, and a 10% solution of potassium cyanide prepared just before use.

Identification of the Naphthols.—0.5 Gram of the substance is placed in a test-tube, and dissolved in the smallest possible quantity of alcohol, added drop by drop. Two c.c. of the copper solution are added, and, after thorough shaking, 4 c.c. of the cyanide. With α -naphthol an abundant, violet-red precipitate is obtained; β -naphthol yields a yellow precipitate. If now just sufficient alcohol is added to dissolve the precipitate, a rose-coloured liquid showing a violet reflection will be obtained with the α -compound, but a golden-yellow solution in the case of β -naphthol.

Identification of the Camphorated Naphthols.—A thread of the

substance is placed in a test-tube, and 2 c.c. of copper solution are added. The substance, which floats on the surface, is dissolved by cautious addition of alcohol, and 4 c.c. of the cyanide are then added. The result is the same as with the ordinary naphthols.

The same process serves for the detection of small proportions of α -naphthol in the β -compound. Benzonaphthol does not give the reaction.

L. DE K.

Estimation of Lactose by Ammoniacal Copper Salt Solutions. YOSHITAKA SHIMIDZU (*Biochem. Zeitsch.*, 1908, 13, 243—261).—Lactose can be estimated by means of the Kumagawa-Suto modification of Pavy's method, if it is first hydrolysed into dextrose and galactose. For the latter purpose, 10% sulphuric acid, or 5% hydrochloric acid, are suitable, and inversion is complete within one hour if 100 c.c. of acid is used for 0.25 to 1 gram of the sugar. The method is applicable to the estimation of sugar in milk.

S. B. S.

Precipitation of Lævulose by Basic Lead Acetate. H. C. PRINSEN-GEERLIGS (*Zeitsch. Ver. deut. Zuckerind.*, 1908, 932—936).—The results of the experiments recorded show that, whilst basic lead acetate does not precipitate lævulose from its solution when the latter contains nothing but the sugar, an appreciable quantity of the sugar is precipitated if other substances which yield insoluble lead compounds are also present. As all syrups and crude sugars give such solutions, these, when clarified with basic lead acetate, furnish filtrates containing less lævulose than the original solution. The reducing sugar precipitated is shown to be actually lævulose.

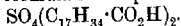
W. P. S.

Detection of Formic Acid in Honey. TH. MERL (*Zeitsch. Nahr. Genussm.*, 1908, 16, 385—389).—Formic acid is best detected in honey by heating the sodium formate, obtained on evaporating the neutralised distillate of the honey, with concentrated sulphuric acid. The carbon monoxide produced is collected over potassium hydroxide solution, and, if its volume is measured, the quantity of formic acid present may be calculated. Lactic acid, which may also be present in the distillate, yields carbon monoxide on treatment with sulphuric acid, and a portion of the distillate should therefore be oxidised with permanganate and the resulting oxalic acid estimated. The quantity of carbon monoxide due to the lactic acid is then deducted from the total volume of gas found. The calomel formed by the action of formic acid on mercuric chloride may also be employed as a measure of the amount of formic acid present, but it is pointed out that other substances present in honey distillates reduce mercuric chloride.

W. P. S.

Acidification and Distillation of Fatty Acids; Distilled Oleic Acid. HUGO DUBOVITZ (*Chem. Zentr.*, 1908, ii, 545; from *Seifensieder Zeit.*, 1908, 35, 728—729).—The acidification of the fatty acids yields, according to Benedikt, sulphostearic acid, which on boiling with water yields hydroxystearic acid. The author, however, obtained about twice the

quantity of hydroxystearic acid than would be expected, and has therefore altered Benedikt's formula for sulphostearic acid into



The presence of small quantities of neutral fat in commercial oleic acid ("distillation olein") is difficult to confirm, owing to the possible presence of anhydrides and lactones. Owing to the presence of volatile fatty acids, the estimation of water in the oleic acid should be effected in a vacuum over calcium chloride.

L. DE K.

Estimation of β -Hydroxybutyric Acid in Urine. PHILIP A. SHAFFER (*J. Biol. Chem.*, 1908, 5, 211—224).—Previous methods for the estimation of this acid are criticised. The method proposed is to distil with sulphuric acid and potassium dichromate, when under suitable conditions, the acid is converted quantitatively into acetone, which is then determined by standard iodine and thiosulphate solutions. A number of precautions, especially in estimations in urine, have to be taken.

W. D. H.

Detection and Estimation of β -Hydroxybutyric Acid in Urine. OTIS F. BLACK (*J. Biol. Chem.*, 1908, 5, 207—210).—Acetoacetic acid is recognised by the red colour produced by ferric chloride. If β -hydroxybutyric acid is tested in the same way in the presence of hydrogen peroxide, the same colour reaction is obtained. In applying the test to urine, evaporation at a gentle heat gets rid of acetoacetic acid; the residue is acidified with hydrochloric acid, and made into a paste with plaster of Paris. This is broken up and extracted with ether, the ether is evaporated, and the test applied to the residue. For quantitative purposes, the residue is extracted with water and examined with a polarimeter.

W. D. H.

Natural and Added Free Tartaric Acid in Natural Wines. H. ASTRUC and J. MAHOUX (*Bull. Soc. chim.*, 1908, [iv], 3, 840—845; *Ann. Chim. anal.*, 1908, 13, 307—315).—The method of estimating the total tartaric acid and the total potassium in a wine, and deducing therefrom the amount of free tartaric acid, gives too low a value for the latter, since the amount of potassium combined with other acids is neglected. In this manner, excessive (added) quantities of free tartaric acid in natural wines are overlooked.

The authors have estimated the total tartaric acid and potassium by the official methods, and the added tartaric acid and total potassium by other methods, in three natural wines prepared by the authors, both when untreated and after addition of variable known quantities of tartaric acid and potassium, and draw the following conclusions from their results. The official methods are liable to large errors in opposite directions, the other methods (Pasteur, Reboul, Magnier de la Source, &c.) are more trustworthy, and should therefore be preferred; it is also more accurate to ignore the estimation of total potassium, and to evaporate in the presence of potassium bromide, in order to estimate free tartaric acid. It is impossible to distinguish natural from added tartaric acid in potable wines.

The authors' results contradict the statement that natural wines from ripe grapes are necessarily always free from tartaric acid.

E. H.

Method of Estimating Succinic Acid in Fermented Liquids containing Other Fixed and Volatile Acids. M. EMMANUEL POZZI-ESCOT (*Compt. rend.*, 1908, 147, 600—601; *Bull. Assoc. chim. Sucr. Dist.*, 1908, 26, 185—186 *).—The following method is stated to be trustworthy for the separation and estimation of succinic acid in solutions containing colouring matters, tannin, proteins, and fixed and volatile acids. A portion of the solution is treated with a small quantity of gelatin, neutralised with ammonia, and then rendered distinctly acid with acetic acid. Barium chloride is next added, and the precipitate, consisting of proteins, tannin, barium oxalate, barium sulphate, &c., is removed by filtration. The filtrate is boiled and treated with a considerable excess of lead acetate, which precipitates any remaining protein and tannin, and phosphoric, tartaric, and citric acids. The precipitate is collected on a filter and washed with very dilute acetic acid. The filtrate is treated with hydrogen sulphide to remove the excess of lead, and the solution is then acidified with sulphuric acid, boiled, and the malic acid is oxidised by the addition of an excess of potassium permanganate. This excess is decomposed by means of potassium hydrogen sulphite, and the sulphuric acid is removed by treatment with barium chloride. The solution is then concentrated, rendered slightly ammoniacal, and the succinic acid is precipitated by the addition of an alcoholic solution of barium bromide, a quantity of alcohol equal to three times the volume of the total solution being also added. The barium succinate is collected on a filter, washed with 60% alcohol, and ignited. The amount of succinic acid is calculated from the weight of barium carbonate obtained (compare this vol., ii, 904).

W. P. S.

Naphtharesorcinol as a Reagent for Certain Aldehyde and Keto-acids. JOHN A. MANDEL and CARL NEUBERG (*Biochem. Zeitsch.*, 1908, 13, 148—151).—The reaction described by B. Tollens for glycuronic acid (heating with naphtharesorcinol and hydrochloric acid, and subsequent extraction with ether) is applicable to several other substances, especially those containing certain combinations of carboxyl and carbonyl groups in the molecule. Also, certain more complex substances, such as glucothionic acid, ovomucoid, chondroitinsulphuric acid, and various nucleo-proteins, give the reaction.

S. B. S.

Separation of Benzoic Acid and Cinnamic Acid. ANNE W. K. DE JONG (*Pharm. Weekblad*, 1908, 45, 1115—1116).—Schering's process (separation of the bulk of the cinnamic acid as calcium cinnamate and oxidation of the remainder with permanganate: *Abstr.*, 1907, ii, 823) is, in the author's opinion, unsuitable for quantitative purposes, owing to the formation of benzoic acid.

L. DE K.

* and *Ann. Chim. anal.*, 1908, 13, 439—440.

Süchting's Method for Estimating Acidity of Soils. BR. TACKE and H. SÜCHTING (*J. pr. Chem.*, 1908, [ii], 78, 139—142).—Mainly polemical. A reply to van Schermbeck (this vol., ii, 743).
J. J. S.

Humic Acid. A. J. VAN SCHERMBECK (*J. pr. Chem.*, 1908, [ii], 78, 285—288. Compare this vol., ii, 743).—A reply to Tacke and Süchting (preceding abstract), and a further criticism of these authors' methods.
G. Y.

Colour Reactions of Sesamé Oil with (a) Aromatic Aldehydes and (b) Various Sugars. The Similar Reactions with Biliary Acids. C. FLEIG (*Bull. Soc. chim.*, 1908, [iv], 3, 984—991, 992—999).—The suggestion has been made (Mylus, *Abstr.*, 1887, 1149) and denied (Ville, *Abstr.*, 1907, ii, 913) that the red or violet colorations formed in the Pettenkofer test for biliary acids, and in the Camoin-Baudouin test for sesamé oil, depend on the formation of furfuraldehyde by the action of the sulphuric or hydrochloric acid on the sucrose used. The author has therefore tried to obtain definite data on this point by using (1) various aromatic aldehydes and (2) polyhydric alcohols and sugars in place of sucrose, and the results, on the whole, support Mylius' contention. It is suggested that the differences in colour produced in using furfuraldehyde instead of sucrose, as noted by Ville, may be due to secondary influences exerted by other hydrolytic products formed when sucrose is used.

Of the thirteen aldehydes tried, *p*-hydroxybenzaldehyde, anisaldehyde, protocatechualdehyde, vanillin, piperonal, and cinnamaldehyde give colours as intense as those yielded by furfuraldehyde, whilst benzaldehyde, salicylaldehyde, *o*-nitrobenzaldehyde, and *p*-dimethylaminobenzaldehyde yield less intense tints, and *m*- and *p*-nitrobenzaldehyde and cuminaldehyde are still less satisfactory.

The polyhydric alcohols and sugars tried as substitutes for sucrose in the Camoin-Baudouin reaction fall into the following five groups, in order of decreasing intensity of colour produced: (a) *lævulose*, invert sugar, sucrose, sorbose; (b) raffinose; (c) xylose, arabinose; (d) galactose, mannose, mannitol, dextrose; (e) *isodulcitol*, erythritol, sorbitol, dulcitol, lactose, maltose, glycerol. All the sugars in this list, except arabinose, were also tried in the Pettenkofer test, and gave similar results.

In both tests the production of colour is expedited by warming the mixture slightly; hydrochloric acid may be used in place of sulphuric acid or vice versa, but in some cases the former acid is advantageous. In the tests for sesamé oil, an alcoholic extract of the oil may be used in place of the oil itself. Full details are given in the two papers as to the exact methods employed in making the tests. The second paper concludes with a summary of the literature dealing with the constituent of sesamé oil to which the production of the colour is due, and the conclusion is drawn that this coloration may be due to the presence of a number of different substances.
T. A. H.

Formation of Formaldehyde in Solutions of Sugar. A. ALEXANDER RAMSAY (*J. Roy. Soc., New South Wales*, 1907, 41, 172—175).—Attention is called to the fact, noticed amongst others by the

author, that small quantities of formaldehyde are formed during the heating or distillation of saccharine liquids (jams, for instance); thus upsetting the value of some tests recommended for the detection of added formalin.

L. DE K.

Identification of Thujone in Liqueurs. LOUIS DUPARC and A. MONNIER (*Ann. chim. anal.*, 1908, 13, 378—382).—The liqueur is submitted to distillation, and 10 c.c. of the distillate (which should be of 60° alcoholic strength) is mixed with 2 c.c. of 10% zinc sulphate, 0.5 c.c. of 10% sodium nitroprusside, and then with 4 c.c. of 5% sodium hydroxide free from carbonate. After the lapse of one minute, 2 to 3 c.c. of glacial acetic acid are added, when, after a few minutes, a very characteristic, dark red precipitate forms if thujone is present.

The author has examined altogether fifty-four different essences (2 parts dissolved in 1000 parts of alcohol), and, with the exception of thujone, they yielded white or yellow precipitates; those which contained citral, however, gave an orange-red deposit.

L. DE K.

Analysis of Scammony Resins. P. GUIGUES (*Bull. Soc. chim.*, 1908, [iv], 3, 872—878).—The ether assay of scammony resin advocated by the *Codex Medicamentarius* is unsound, since (1) the resin of jalap is partly soluble in ether, whilst some resins from scammony are incompletely soluble in this solvent; (2) it permits the substitution of fusiform jalap resin, soluble in ether, for the true scammony resin; (3) it prevents the recognition of foreign resins, particularly colophony, soluble in ether; and (4) it is subject to numerous errors, and does not give constant results.

The solubility of scammony resin is not appreciably altered by boiling with water or with 10% hydrochloric acid for several hours. Assay of the resin by turpentine instead of ether is no more successful. The author proposes to detect adulteration by determination of the rotatory power. The following are the values obtained for the optical activities of resins: Commercial Tampico jalap, $-34^{\circ}20'$; true Orizaba jalap, $-24^{\circ}45'$; officinal jalap, -36° ; officinal turbith, $-30^{\circ}10'$ to $31^{\circ}35'$; ordinary colophony, $+6^{\circ}$ to $+7^{\circ}$; sandarac from the Arabic bazaar, $+46^{\circ}20'$; pure sandarac, $+31^{\circ}$ to $+34^{\circ}$; recent mastic, $+29^{\circ}30'$; second quality mastic, $+21^{\circ}50'$; guaiacum, -17° . For scammony, the author obtained $-24^{\circ}30'$ as a maximum from the resin extracted from the gum-resin, and $-18^{\circ}30'$ to $-23^{\circ}30'$ for resins extracted from the roots. The rotatory power is independent of the solubility in ether. These values show that only the Orizaba jalap (fusiform) resin and guaiacum resin can be used as adulterants. Since only the scammony resins extracted from the gum resin, which are at least twice as expensive as that extracted from the root, have rotatory powers as high as $-24^{\circ}30'$, a resin having α_D higher than $-23^{\circ}30'$ is most probably fusiform jalap resin. Guaiacum resin should be readily detected by its chemical characteristics.

E. H.

Detection of Arbutin in Plants. MILLE A. FICHTENHOLZ (*J. Pharm. Chim.*, 1908, [vi], 27, 255—262).—In applying Bourquelot's method (*Abstr.*, 1902, ii, 55) to the detection of glucosides in plants,

it is necessary to allow the emulsin to act on the plant extract during several days, as the action of the ferment is frequently slow, owing to the presence of tannic or gallic acids, which retard its activity. In the case of such glucosides as arbutin, which has a high levorotation, it is advantageous to defecate the plant extract with lead acetate before adding emulsin. Bearberry leaves contain at least 1.66% of arbutin.

T. A. H.

Volumetric Estimation of Potassium Ferrocyanide. H. BOLLENBACH (*Zeitsch. anal. Chem.*, 1908, 47, 687—690).—The following modification of de Haën's process was found to give trustworthy results. The potassium ferrocyanide solution is acidified with sulphuric acid, and an excess of *N*/10 permanganate solution is added. After the addition of a few drops of ferric sulphate solution, the excess of permanganate is titrated with *N*/10 ferrocyanide solution. Each drop of the latter added produces a greenish-blue precipitate, which disappears on shaking, as long as any permanganate remains.

W. P. S.

Detection and Estimation of Hexamethylenamine in Pharmaceutical Mixtures. WILLIAM A. PUCKNER and W. S. HILPERT (*J. Amer. Chem. Soc.*, 1908, 30, 1471—1474).—The process is based on the fact that hexamethylenamine does not suffer decomposition on boiling with aqueous potassium hydroxide, but that it is converted into formaldehyde and ammonium sulphate on boiling with dilute sulphuric acid. The ammonia formed is then readily estimated, any ammonia pre-existing in the sample under examination being removed by the preliminary alkaline treatment.

The reactions with mercuric chloride and with bromine are also useful in the identification of hexamethylenamine. Advantage may also be taken of its solubility in chloroform.

L. DE K.

Estimation of the Total Alkaloids in Cinchona Barks. N. H. COHEN (*Pharm. Weekblad*, 1908, 45, 1089—1098).—A modification of Florence's method (*Abstr.*, 1907, ii, 317). Five grams of the very fine powder are placed in a small corked flask, 125 grams of ether and 5 c.c. of 15% sodium hydroxide are added, and the whole is weighed. After shaking vigorously every five minutes for an hour, the flask is fitted to a vertical condenser, and the contents are boiled for half an hour. When cold, ether is added to restore the original weight, 5 c.c. of water are introduced, and, after thorough shaking, the ether is transferred to a 250 c.c. flask. Twenty c.c. of lime water are added, the flask is closed, preferably with the same cork, and the whole thoroughly shaken. One hundred c.c. of the ether are now drawn through a plug of cottonwool into a weighed 200 c.c. flask, the ether is distilled off, and the residual alkaloids dried for one and a-half hours at 100° and weighed. The weight $\times 25 =$ percentage of alkaloids, about 0.5 of which consists of impurities. These may be estimated by dissolving the alkaloids in 10 c.c. of 2% sulphuric acid and shaking the solution with ether, which is then evaporated in a weighed flask.

Florence's estimation of the quinine by means of *N*/10 ethereal oxalic acid cannot be recommended.

L. DE K.

The Quantitative Relations of the Thalleoquinine Reactions.

JOS. VONDRASEK (*Chem. Zentr.*, 1908, ii, 833—834; from *Pharm. Post*, 1908, 41, 605—607).—The author recommends that the test for quinine should be carried out as follows: 0.01 gram of the quinine salt is dissolved in a mixture of 1 c.c. of *N* hydrochloric acid and 2 c.c. of *N*/10 potassium bromate, and boiled until the liquid turns orange-yellow. When cold, 1—2 c.c. of ammonia are added, and a dark green solution is obtained. Quinidine also gives the test. In this reaction, three atoms of chlorine are taken up by 1 mol. of quinine. The test may be also carried out in the cold, when addition of ammonia will give a dark green precipitate and a dark green liquid. On adding alcohol, a solution is obtained which is suitable for the colorimetric estimation of quinine.

Quinine and quinidine may be distinguished from cinchonine and cinchonidine by heating the aqueous solutions with excess of potassium bromate, which yields a blue liquid. The colour is destroyed on adding acid.

L. DE K.

Assay of Coca. ANNE W. K. DE JONG (*Chem. Weekblad*, 1908, 5, 645—647).—A reply to M. Greshoff (this vol., ii, 441). The author gives experimental results in support of the contention that Greshoff's method of estimating coca is untrustworthy, and attributes this to decomposition caused by heating the alkaloid salts in aqueous solution (compare Abstr., 1905, ii, 778; this vol., ii, 440).

A. J. W.

Assay of Coca. MAURITS GRESHOFF (*Chem. Weekblad*, 1908, 5, 705—706. Compare this vol., ii, 441).—Polemical. A final reply to de Jong (preceding abstract).

L. DE K.

Detection of Vegetable Poisons in Decomposed Animal Bodies. THEODOR PANZER (*Zeitsch. anal. Chem.*, 1908, 47, 572—590).—Corpses which have undergone a considerable amount of decomposition may contain substances which yield general reactions similar to those given by the alkaloids. These substances, which are chiefly found in the brain, and to a less extent in the liver and kidneys, do not give the characteristic reactions of the separate alkaloids. If a residue is obtained from the ethereal extract of the alkaline solution obtained in the usual way (Stas-Otto method) from the organs under examination, which residue gives general alkaloidal reactions, the solution of the residue should be rendered acid with hydrochloric acid and extracted with ether; this treatment removes the substances, and the aqueous solution is then examined for alkaloids. If possible, it is preferable not to mix the brains with the other organs of the body under examination.

W. P. S.

Process for the Valuation of Saffron. BALTHASAR PFYL and W. SCHEITZ (*Zeitsch. Nahr. Genussm.*, 1908, 16, 347—352).—The

process depends upon the estimation of the amount of sugar formed by the hydrolysis of the glucoside soluble in chloroform, which the authors have shown occurs in saffron (this vol., ii, 979). Saffron is dried, powdered, and re-dried, and 5 grams of the preparation extracted in a Soxhlet apparatus with light petroleum and, after drying, re-extracted with dry chloroform. The solvent is evaporated from the chloroformic extract, and the residue dissolved as far as possible in acetone. The latter is evaporated, and the glucoside in the residue hydrolysed by adding 5 c.c. of *N*-hydrochloric acid and heating during fifteen minutes, water being added as required to bring the total amount of liquid up to 25 c.c. The filtrate from this is neutralised with *N*-alkali, and the sugar estimated by Allihn's method.

The best quality of commercial saffron, consisting only of the stigmas of the saffron crocus, yields in this way 0.1996 to 0.2090 gram of metallic copper, and samples yielding less than this may be regarded as having an unduly large proportion of styles included, or as containing constituents foreign to the saffron crocus. The quantity of adulterant present is not directly proportional to the diminution in the amount of copper obtained, since the latter is, in part, dependent on the concentration of sugar solution used, and a table is supplied in the original from which, by a method of interpolation, the quantity of "real saffron" present in a sample can be calculated from determinations made by the above process. Trials with the usual adulterants of saffron, namely, saffron crocus styles, logwood, red poppy petals, red peony petals, honey, marigold petals, safflower, Cape saffron, and Spanish thistle flowers, show that each of these products, when subjected to this process, yields practically no reducing substance, and that with turmeric and red sandalwood very much smaller quantities of copper are obtained than with real saffron.

T. A. H.

Detection of Proteins by means of Formaldehyde. LEO VON LIEBERMANN (*Zeitsch. Nahr. Genussm.*, 1908, 16, 231).—Five c.c. of the solution to be tested are treated with one drop of 40% formaldehyde solution and one drop of very dilute ferric chloride solution. The mixture is then allowed to flow over the surface of 5 c.c. of concentrated sulphuric acid. If protein (albumin or albumose) is present in the solution, a violet ring is observed at the junction of the two liquids.

W. P. S.

A New Reaction for Bile Acids. ADOLF JOLLES (*Zeitsch. physiol. Chem.*, 1908, 57, 30—34; *Ber.*, 1908, 41, 2766).—If a few drops of a 5% solution of rhamnose are warmed with a similar amount of a 0.1% solution of taurocholate or glycocholate, and a few drops of concentrated hydrochloric or sulphuric acid, a momentary red coloration is obtained, which passes in a short time into a green fluorescence. This test is given also by cholic acid, but not by glycine or taurine. The delicacy of the test is very considerable. In employing it to detect bile acids in urine, the fluid is first mixed with a solution of caseinogen, and the latter is precipitated by careful

addition of sulphuric acid and filtered off. The precipitate is digested with absolute alcohol at room temperature for an hour, filtered, and the reaction tried with the filtrate. Cholesterol, albumin, urea, carbohydrates, higher hydrocarbons, and acids of the fatty and benzene series do not give the test.

W. D. H.

A Colour Reaction of Bile Acids with Vanillin and Sulphuric Acid. KATSUJI INOUE and HIIZU ITO (*Zeitsch. physiol. Chem.*, 1908, 57, 313—314).—If vanillin is added to an aqueous solution of bile acids, and then concentrated sulphuric acid, a red ring appears at the junction of the two liquids; on shaking up the mixture, it becomes red, then brown or yellow, and finally violet. On diluting this with glacial acetic acid, a wide absorption band at *D*, reaching to *C* on one side and *E* on the other, is seen. Tables are given showing the delicacy of the test with cholic, glycocholic, and taurocholic acids.

W. D. H.

The Lecithin-content of Milk. JOSEPH NERKING and E. HAENSEL (*Biochem. Zeitsch.*, 1908, 13, 348—353).—For estimation of lecithin, 100 c.c. of milk were precipitated with 200 c.c. of alcohol. The precipitate was extracted with chloroform in a Soxhlet apparatus. The alcoholic filtrate was evaporated at 50—60°, and the residue also extracted with chloroform. The two chloroform extracts were united, and the residue, after evaporating off the solvent, incinerated with fusion mixture. The amount of phosphoric acid in the ash was estimated, and from the results, the lecithin-content calculated. The amount of lecithin from several species of animals varied (as a mean of several analyses in each case) between 0.0109 and 0.0833%.

S. B. S.

Detection of Indican in Urine. ERNST SALKOWSKI (*Zeitsch. physiol. Chem.*, 1908, 57, 519—521).—The urine is mixed with copper sulphate solution and hydrochloric acid, and then chloroform added; the chloroform is coloured blue if indican is present. This method is described with full details, as to procedure and the precautions to be observed.

Gautier's statement that the urine of rabbits fed on cabbage does not contain indican is incorrect.

W. D. H.

The Guaiacum Reaction. CARL L. ALSBERG (*Arch. exp. Path. Pharm., Suppl.*, 1908, 39—53).—Various points in relation to this test for blood are discussed, and its unsatisfactory nature pointed out. The blood of many invertebrates gives the reaction, and particular attention has been paid to blood which contains hæmocyanin. This blood gives the test even after it is boiled. Hæmocyanin itself, other copper compounds, certain manganese compounds, and other salts give the test.

W. D. H.

Estimation of Catalases and Oxydases in Blood. I. WALTHER LOB (*Biochem. Zeitsch.*, 1908, 13, 339—347).—A preliminary account is given of the apparatus employed for the estimation of the amount of oxygen evolved when a hydrogen peroxide solution is treated with blood. The gas evolved was measured both by a "volume method"

and a "pressure method." In the former case, the vessel containing the peroxide was connected with a Hempel burette, and in the latter, with a manometer. The blood in both cases was contained in a small capsule resting on a plate which was fixed on to a ground stopper inserted into the side of the peroxide containing vessel above the level of the liquid. By turning the stopper through 180° , the capsule could be dropped into the peroxide solution. This was only done when the apparatus was in connexion with the burette or manometer, and had reached the constant temperature at which the experiment was carried out.

S. B. S.

Diazo-reaction of Atoxyl. ERCOLE COVELLI (*Chem. Zeit.*, 1908, 32, 1006).—Solutions of atoxyl give the following reactions: (1) with a hypochlorite, a yellow precipitate or coloration; (2) with a hypochlorite, phenol, and ammonia, a blue coloration, which can be observed even in a dilution of 1 : 100,000; (3) with reducing agents, such as zinc and sulphuric acid in the cold, or stannous chloride or hypophosphorous acid in hot hydrochloric acid, a yellow precipitate; and (4) with a fatty aldehyde and a hot mineral acid, a yellow coloration.

More delicate and characteristic reactions are given by arsenodiazobenzene, which is formed when atoxyl is treated with a few drops of a 1/2% solution of sodium nitrite and a few drops of sulphuric acid. The resulting solution gives: (a) with α -naphthylamine hydrochloride, a purplish-red coloration. This extremely delicate reaction is recommended for the detection of unchanged atoxyl, for example, in urine. Carbamide and the amino-acids, which react with nitrous acid, do not interfere with the test. The reaction may be employed also for the colorimetric estimation of atoxyl; (b) with a few drops of acet-aldehyde and potassium hydroxide, added drop by drop, a carmine-red coloration, changing to yellow, and finally becoming colourless. The carmine-red is stable in presence of an excess of alkali; and (3) with phenols, such as β -naphthol, abrastol, resorcinol, morphine, or dinine, a purplish-red coloration. Towards biliverdin and pathological urines, which give Ehrlich's diazo-reaction, arsenodiazobenzene behaves in the same manner as sulphodiazobenzene.

G. Y.

General and Physical Chemistry.

Refractive Indices of Solutions. FREDERICK H. GETMAN and F. B. WILSON (*Amer. Chem. J.*, 1908, 40, 468—484).—The specific refraction of various salts, acids, and non-electrolytes in water, calculated on the assumption that the refractive powers of solvent and solute are uninfluenced by the act of solution, have been compared with their specific refractions calculated from atomic refractions.

The lack of agreement in most of the cases studied is supposed to indicate the presence of hydrates in solution, but it has not been found possible to calculate the degree of hydration from the results.

R. J. C.

Refractometric Studies of some Methane Derivatives in which Two or Three Atoms of Hydrogen are Replaced by Negative Radicles. II. Sodium Salts. ALBIN HALLER and PAUL TH. MÜLLER (*Ann. Chim. Phys.*, 1908, [viii], 15, 289—296).—Mainly a résumé of work already published (*Abstr.*, 1905, i, 112); the difference between the values of M_D for free acid and sodium salt in the case of ethyl cyanoacetoacetate is 3.74; the acid therefore is a *pseudo-acid*. The possible constitutional formulæ of the sodium salts of the *pseudo-acids* are discussed.

M. A. W.

Atomic Decomposition and Spectral Series. AUGUST L. BERNOULLI (*Physikal. Zeitsch.*, 1908, 9, 745—749; *Ber. deut. physikal. Ges.*, 1908, 6, 636—642).—The author supposes that the molecules of a gas are not alike, but that the molecular and atomic weights represent average values. On the assumption that every chemical element in the gaseous condition represents a conglomeration of polymerised forms of a primordial matter in equilibrium, and that the gas mixtures obey Rayleigh's radiation law, a formula is deduced from which the atomic weights of the elements can be calculated. The formula is written

$$A = 1.0104 \left(\frac{-1 - \sqrt{3}}{2} \right)^p \cdot \left(\frac{-1 + \sqrt{3}}{2} \right)^q,$$

p and q being integral numbers. Using different values of p and q , atomic weight numbers in very good agreement with the experimental values are obtained.

Similar assumptions to the above have led the author to a formula which gives the lines and bands in the spectra of the elements with considerable accuracy.

H. M. D.

Flame Spectrum of Cupric Chloride. PETER KIEN (*Zeitsch. wiss. Photograph. Photophysik. Photochem.*, 1908, 6, 337—358).—The first part of the paper contains an historical review of previous investigations on the spectrum of cupric chloride; in the second, new experiments are described, the object of which was to determine the intensity and the structure of the bands.

The cupric chloride was introduced into the flame of a blow-pipe

fed with coal-gas and air, or oxygen, by means of a roll of filter paper moistened with a solution of the salt. By means of a spring and clockwork mechanism, the filter paper roll was moved up into the flame at a definite rate, and a steady supply of cupric chloride thereby obtained.

The intensity of the spectrum is not greatly influenced by the temperature, but depends very much on the amount of cupric chloride in the flame. As already pointed out by Lecoq de Boisbaudran, four stages of the spectrum, depending on the amount of salt in the flame, can be distinguished. The author's observations agree very well with those of Boisbaudran, except in the fourth stage, when an extremely small amount of salt is present; in these circumstances the only remaining band of any considerable strength reaches from $\lambda = 5100$ to $\lambda = 5800$, and has a maximum at $\lambda = 5400-5600$.

The four characteristic double bands which are seen in the second stage: α ($\lambda = 4259-4279$), β ($\lambda = 4333-4353$), λ ($4412-4433$), and δ ($4493-4515$), are found with great dispersion to consist of groups of bands in which series of lines can be recognised.

Attention is called to the sensitiveness of the cupric chloride spectrum. Photographic registration of the "sodium chloride" spectrum in coal or coke fires has confirmed Salet's conclusion that this is due to traces of cupric chloride.

H. M. D.

Spectrum of Neo-erbium Oxide and Kirchhoff's Law. KARL A. HOFMANN and GÜNTHER BUGGE (*Ber.*, 1908, 41, 3783-3789, Compare Hofmann and Burger, this vol., ii, 189).—The reflection spectrum of neo-erbium oxide illuminated by an electric arc light is far more characteristic than the absorption spectrum of a 10% solution of the nitrate, and, in agreement with Kirchhoff's law, it is found that the lines of maximum absorption in the former spectrum correspond with the bright lines in the emission spectrum of the oxide heated with a hydrogen flame. The bands of maximum absorption of the nitrate solution do not exactly correspond with the lines of the oxide spectrum, but are somewhat displaced; thus, the maxima of emission of the oxide are: $\lambda = 661.3, 653.0, 563.4, 540.2, 522.6, 493.0, 489.1, 463.0$, and 447.0 ; the maxima of absorption of the oxide are: $\lambda = 661.7, 652.4, 563.8, 540.4, 522.4, 491.5, 489.5, 462.5$, and 446.5 ; and of the nitrate solution, $\lambda = 667, 654, 541, 523, 519, 492, 487, 450$, and 442 . Both the oxalate and fluoride in a solid state give absorption spectra very similar in character to the absorption spectra of solutions of the nitrate and sulphate, although the intensities and positions of the bands vary slightly in each case. Sharp lines as well as bands appear in the absorption spectrum of the solid sulphate, so that a transition of the band spectrum of a salt to the line spectrum of the oxide appears probable.

W. H. G.

Spectroscopic Behaviour of Hydrocarbons with Conjugate Ethylene Linkings. JULIUS W. BRÜHL (*Ber.*, 1908, 41, 3712-3720, Compare Trans., 1907, 91, 115).—Reif's results (this vol., i, 847) confirm the author's previous conclusions. The values for the refractive dispersions Δ^{B^6} -hexenol and Δ^{B^6} -hexadiene have been re-determined and Reif's results confirmed.

In the latter compound, the values indicate exaltations of +1.49, +1.71, and +0.52 units in the values for $(n_a^2 - 1)/(n_a^2 + 2) \times P/d$, $(n_b^2 - 1)/(n_b^2 + 2) \times P/d$, and $M_\gamma - M_\alpha$ respectively.

Using the formula $(n^2 - 1)/n^2 + 2 \times P/d$, the following values have been calculated for $\Delta\gamma$ -hexatriene from W. H. Perkin's numbers:

H_α	D.	$M_\gamma - M_\alpha$
30.59	31.03	2.22

which indicate exaltations of 2.07, 2.49, and 1.01 units respectively. These results are striking when compared with the normal optical behaviour of benzene, $\Delta^{1,3,5}$ -cyclohexatriene, which shows practically no exaltation. Harries and Majima's $\Delta^{1,2}$ -dihydrocymene (this vol., i, 733) also exhibits characteristic exaltations, due to conjugated ethylene linkings.

It is pointed out that the intermediate product described by Auwers and Hessenland (this vol., i, 551) in the preparation of $\Delta^{1,3}$ -dihydro-*p*-xylene, and stated to be the corresponding acid, cannot have this constitution, as its methyl ester exhibits no exaltation, but is optically normal. It is suggested that the ethylene linkings in the acid are not in conjugate positions.

Zelinsky and Gorsky (this vol., i, 619) claim to have prepared dihydrobenzenes which give normal molecular refractions and dispersions, although they contain conjugated ethyl linkings. It is pointed out that these authors have not conclusively proved the constitution of their compounds by the examination of decomposition products. J. J. S.

Water of Crystallisation as Affected by Light. RALPH H. MCKEE and ELVIN J. BERKHEISER (*Amer. Chem. J.*, 1908, 40, 303—305).—Aniline-*p*-sulphonic acid has been found to lose its water of crystallisation more readily when exposed to light than when kept under exactly similar conditions in the dark. Careful observations showed that, although the temperature of the specimens exposed to direct sunlight was 2.86° lower than those screened from such light by black paper, yet the former lost more than 98% of their water of crystallisation during ten winter days' exposure, whilst the latter only lost 35% during the same period. From determinations of the solubility of the product, no evidence was obtained of any transformation having taken place into the ortho- or meta-compound. When hydrated sulph-anilic acid or the product obtained after exposure to sunlight was crystallised from water above 80°, it was found to be in an anhydrous state, whilst from water at 40° the crystals obtained had again 2H₂O. J. V. E.

Violet Coloration Produced in Glass by the Influence of Light. J. G. MASCHHAUPT (*Chem. Weekblad*, 1908, 5, 807—808).—The author attributes the violet colour sometimes developed in the glass of old mirrors to the action of sunlight, and describes experiments in support of his contention. A. J. W.

Some Reflections on Radiology. ALBERT REYCHLER (*Bull. Soc. chim.*, 1908, [iv], 3, 1009—1012).—Rutherford and Soddy's theory that radioactive substances consist of unstable, large accumulations of particles of a primordial matter, which are liable to internal

changes and decompositions, tending towards closer and more stable arrangements of the particles, does not in the author's view account for all the facts observed, and particularly for (a) the large amounts of energy disengaged and (b) the conversion of an atom of uranium into several atoms of helium (f) and one atom of radium, which is more voluminous and more active than that of the parent substance.

These difficulties disappear if the ether is taken as the primordial matter, and is regarded as existing in elementary atoms in two forms, namely, condensed, as nodules, an accumulation of which forms the atom, and also in the free state. If the intra-atomic transpositions may then be regarded as involving also the condensation of the free ether, the changes which occur can be represented thus: radium + ether = α -particles + β -particles + emanation. This view would explain the discrepancy between Ramsay and Rutherford's estimates of the length of "life" of a radium atom, that of the former being too short. It implies, however, that radioactive matter enclosed in a glass tube should gain in weight, since the ether would have free access and only β -rays could escape. The β -rays it is suggested, however, may be in part an inflow of electropositive ether.

T. A. H.

Extraordinary Radioactivity of Water from a Spring at Valdemorillo. JOSÉ MUÑOZ DEL CASTILLO (*Anal. Fis. Quim.*, 1908, 6, 398—400).—Samples of water from a spring at Valdemorillo were found to have radioactivities varying from 27,000 to 51,687 volts per hour-litre. The latter value is remarkably high, and much exceeds that found with the waters from Lerez (10,000—12,000 volts per hour-litre: compare Abstr., 1907, ii, 218; this vol., ii, 750).

W. A. D.

Radioactivity of Three Springs at Oña (Burgos). JOSÉ MUÑOZ DEL CASTILLO (*Anal. Fis. Quim.*, 1908, 6, 400—402).—The following table shows the character of the rock through which the springs percolate, and the radioactivity of the water and mud therefrom in volts per hour-litre:

	Rock.	Activity of water.	Activity of mud.
Spring A	Argillaceous limestone.....	34.8	23.6
„ B	Cretaceous „	30.0	99.3
„ C	Argillaceous „	12.8	30.2

Spring B is one of considerable volume feeding the river Oca. All three springs come to the surface within a radius of 200 metres from one another. It is probable that all three springs arise from a common source; the difference between the values for the mud of B and that from A and C is probably due merely to the muds being of different consistency.

W. A. D.

Radioactivity of the Gases of the Thermal Spring of Uriage (Isère). GUSTAVE MASSOL (*Compt. rend.*, 1908, 147, 844—846).—During the passage of the water through a siphon to the reservoir, very variable quantities of gas are evolved; determina-

tions in two successive half-hours (when a considerable volume of water was flowing) gave 538 litres and 724 litres respectively. Of this gas, 7% is soluble in potassium hydroxide solution (carbon dioxide and hydrogen sulphide), whilst the remainder consists of nitrogen and allied gases. The radioactivity of the gas, as measured by a Curie electroscope (as modified by Chéneveau and Laborde), was 0.150 and 0.144 per 10 litres of gas (at 20° and 745 mm.), the radioactivity of the emanation per minute from 1 mg. of radium being taken as the unit. These results are not strictly comparable with Besson's (following abstract), since the latter refer to the gas dissolved in the water, of which 64.6% is insoluble in potassium hydroxide solution (Lefort found that 1 litre of the water of Uriage contains 3.2 c.c. CO_2 , 7.44 c.c. H_2S , and 19.5 c.c. N_2). For the radioactivity of the gas remaining dissolved in the water, the author obtains the value 0.500 per 10 litres. The saline residue left on evaporating the water, the deposit formed in the reservoir, and the rock from which the water flows are all quite inactive.

E. H.

Radioactivity of the Waters of Uriage-les-Bains (Isère). PAUL BESSON (*Compt. rend.*, 1908, 147, 848—850. Compare preceding abstract).—Three litres of the water were boiled for one hour, the gases evolved being collected over mercury and dried by means of potassium hydroxide and phosphoric acid. After keeping for three hours, the radioactivity of the gas was measured by means of the Chéneveau and Laborde modification of the Curie electroscope. For the gas from 10 litres of water, the values 0.012, 0.015, and 0.018 in mg.-minute units were obtained at pressures of 745 mm., 740 mm., and 735 mm. respectively. Thus the value seems to increase as the atmospheric pressure decreases. This observation of radioactivity in a mineral water containing 6.0567 grams of sodium chloride and 3.311 grams of calcium, magnesium, and sodium sulphates is in accordance with Kofler's work on the solubility of the emanation in saline solutions. The period of half-decay according to Curie's law is four days. The residue left on evaporation is inactive.

The ferruginous water has the very low radioactivity 0.003 mg.-minutes.

E. H.

Absorption of the Radioactive Emanations by Charcoal. R. W. BOYLE (*J. Physical Chem.*, 1908, 12, 484—506).—Experiments have been made to determine how the absorbing power of charcoal for the emanations of radium and thorium depends on the conditions.

The absorbing power of charcoal for radium emanation at the ordinary temperature is considerable, but the rate of absorption soon falls off, owing to saturation of the upper layer and the comparatively slow rate of diffusion to the lower layers.

The majority of the experiments were made with thorium emanation. As the rate of decay of the emanation is so great, the observations were made by leading the latter, mixed with air, through tubes containing various absorbing or non-absorbing materials, and estimating its activity by measuring the ionisation of air produced. The results are given in the form of tables and curves, the latter being

obtained by plotting the ionisation current against the speed of the air current.

The degree of absorption of the emanation depends : (1) on the speed of the air current, being diminished by increasing the speed ; (2) on the nature of the charcoal, being greatest for cocoanut, intermediate for animal, and least for wood, charcoal ; (3) on the extent of absorbing surface exposed, being greatest for the most finely-divided charcoal, and (4) is greater the lower the temperature.

G. S.

Action of the Radium Emanation on Water. ERNEST RUTHERFORD and T. ROYDS (*Phil. Mag.*, 1908, [vi], 16, 812—818. Compare Cameron and Ramsay, *Trans.*, 1908, 93, 992).—Preliminary experiments are described, the object of which was to ascertain the minimum amount of neon which can be detected spectroscopically. Small quantities of air were subjected to the action of cocoanut charcoal, cooled by liquid air, and the residual gas introduced into a small spectrum tube, 7 cm. long and 1 mm. in diameter, and examined for neon. Operating on 1/15 c.c. of air, the yellow line of neon is readily observed ; with 2/15 c.c. of air, the red lines of neon are clearly visible, and with 1/5 c.c. of air, a brilliant neon spectrum showing most of the lines is obtained. Since, according to Ramsay, one volume of neon is contained in about 100,000 volumes of air, these experiments show that less than one-millionth of a c.c. of neon can be detected spectroscopically.

The gases formed by the action on water of a quantity of emanation, corresponding with the equilibrium amount from 150 mg. of radium, were then examined in a similar manner, after the hydrogen and oxygen had been removed by explosion. A complete and brilliant spectrum of helium was found, but there was no trace of a neon spectrum. In all, five experiments were made ; neon was only observed in one case, and was found to be due to a small air leak. The gases obtained from the aqueous solution of the radium salt, from which the emanation had been pumped off, also showed no trace of neon when examined in the same way.

The positive results obtained by Cameron and Ramsay in similar experiments are attributed to the leakage of air into the apparatus used.

H. M. D.

Nature of the Positive Rays. WILHELM WIEN (*Sitzungsber. K. Akad. wiss., München*, 1908, 55—65).—Experiments are described the object of which was to find an explanation for the well-known fact that the canal rays are unequally deviated under the influence of a magnetic field. The results indicate that this lack of uniformity cannot be due to the ions having different masses, but is a consequence of differences in the lengths of the paths which they traverse in the charged condition. The nature of the canal rays is determined by a condition of equilibrium, in which the ratio of the charged to the uncharged particles has a constant value. Under the influence of a magnetic field, some of the charged particles are removed, but the condition corresponding with the equilibrium ratio is immediately restored by dissociation of the atoms.

H. M. D.

β -Rays of Actinium. OTTO HAHN and LISE MEITNER (*Physical. Zeitsch.*, 1908, 9, 697—702).—The β -rays emitted by actinium are not absorbed by aluminium according to an exponential formula. With gradually increasing thicknesses of aluminium, the absorption is at first more rapid than that required by such a formula; in the second stage, the absorption is exponential, whilst in the third, a more rapid rate of absorption is again found. It is found that the increase in the absorption in the third stage is dependent on the experimental arrangements.

The large value of the absorption coefficient for small thicknesses of the absorbing layer is found to be due to the emission of easily absorbed β -rays by radio-actinium. This was separated from the other products of disintegration, and its β -ray activity examined. In addition to β -rays, which are absorbed to the extent of 50% by 0.04 mm. of aluminium, it emits a much more penetrating type of radiation, which consists either of hard β -rays or soft γ -rays.

β -Rays are also emitted by actinium A and actinium C. The former are absorbed even more readily than the soft β -rays of radio-actinium; the latter are absorbed to the extent of 50% by 0.24 mm. of aluminium.

The view that homogeneous products emit homogeneous β -radiation is confirmed by the results of this investigation. H. M. D.

Influence of Pressure on Ionisation Produced in Gases by the X-Rays. The Saturation Current. E. ROTHÉ (*Compt. rend.*, 1908, 147, 785—788).—A method is described for studying the influence of pressure on ionisation phenomena in general, and details are given of the precautions necessary to secure regular working of the Crookes' tube. The author shows that the intensity of the saturation current is proportional to pressure from 0.1 to 5 atmospheres. W. O. W.

Volatilisation Produced by Canal Rays. JOHANNES STARK (*Zeitsch. Elektrochem.*, 1908, 14, 752—756).—The canal rays consist of charged or uncharged atoms or molecules projected with great velocity. When such particles strike a metal, they impart their velocity to one or more atoms of the metal, which may rebound with sufficient violence to pass away from the surface of the metal. The consequences of this idea are followed out, and the author thinks that the cathodic volatilisation of metals is better explained in this way than by Kohlshütter's chemical theory (this vol., ii, 457). T. E.

Electromotive Force of Iodine Concentration Cells in Water and Alcohol. ARTHUR P. LAURIE (*Proc. Roy. Soc. Edin.*, 1908, 28, 382—393; *Zeitsch. physikal. Chem.*, 1908, 64, 615—628).—Measurements are recorded of the *E.M.F.* of concentration cells consisting of platinum wires immersed in differently concentrated solutions of iodine in aqueous and alcoholic potassium iodide.

For aqueous solutions in which the iodine concentration is small, the observed values of the *E.M.F.* of the concentration cell are in good agreement with those calculated from Nernst's equation when

the free iodine concentrations are determined from the equilibrium constant corresponding with the dissociation equation: $KI_a \rightleftharpoons KI + I_2$. For more concentrated solutions, the observed values differ sensibly from those obtained by calculation.

Similar relationships between the two series of numbers are found in the case of alcoholic solutions, and the conclusion is drawn that Nernst's equation also applies to these solutions and that they contain potassium tri-iodide like the aqueous solutions.

Measurements in aqueous alcoholic solutions show that the *E.M.F.* of a given concentration cell increases as the proportion of alcohol in the mixture increases.

When solutions containing equal quantities of iodine and potassium iodide in water and alcohol respectively are opposed to one another, a considerable *E.M.F.* is obtained. The action of the cell involves the transference of iodine from the aqueous to the alcoholic solution, and the transference of potassium iodide in the reverse direction. In both cases the transference is towards the solvent in which the substances dissolve more readily.

H. M. D.

Explanation of Supertension. III and IV. FELIX KAUFER (*Zeitsch. Elektrochem.*, 1908, 14, 737—741, 749—752).—In consequence of Müller's criticism (this vol., ii, 802), the author has made measurements of the difference of potential between lead or smooth platinum cathodes and a solution of acetophenone or benzophenone in aqueous alcohol containing potassium acetate during electrolysis. These show that, under the same conditions, the fall of potential between the solution and the cathode is greater with lead than with platinum.

The resistance of a cell with a mercury cathode and an anode of platinum or of lead in which the electrolyte is sulphuric acid of maximum conductivity is measured during the passage of a current. With a low current density (under 0.05 ampere per sq. cm.) the attainment of a steady condition is extremely slow. The resistance of the cell is much larger than that observed with no current flowing; it is greatest with small current density, and is not dependent on the nature of the anode. Stirring the mercury does not change the resistance. As in the case of lead previously investigated (this vol., ii, 558), the author assumes the existence of a film of a hydride of high resistance on the surface of the mercury. The product of film resistance and current, which is the supertension due to the transition resistance at the surface of the cathode, is approximately independent of the current density and varies from 0.05 to 0.1 volt.

T. E.

Influence of the Velocity of Ionic Reactions on the Current-Potential Curve. ARNOLD EUCKEN (*Zeitsch. physikal. Chem.*, 1908, 64, 562—580).—The relation of current and potential has been studied in the double cyanides of potassium with silver, gold, copper, and mercury, but only the first of these allows of the production of a limiting current independent of the potential. The velocity of dissociation, $Ag(CN)_2 \rightarrow Ag + 2(CN)'$, is very great. The finite

velocity observed by Caspari (Abstr., 1900, ii, 7) is due to a secondary reaction. The smaller the velocity of the ionic reaction the smaller is the limiting current.

The velocity of dissociation of acetic, lactic, and benzoic acids has been calculated from the results of previous observers. C. H. D.

Ionisation Constants of the Secondary Hydrogen Ion of Dibasic Acids. RUDOLF WEGSCHEIDER (*Zeitsch. Elektrochem.*, 1908, 14, 740-741).—A claim of priority against Chandler (this vol., ii, 467). T. E.

Specific Charge of the Ions Emitted by Hot Substances. OWEN W. RICHARDSON (*Phil. Mag.*, 1908, [vi], 16, 740-767).—The ratio of the electric charge to the mass of the positive ions emitted by hot substances has been determined by measurements of the deflexion of the path, due to the action of a transverse magnetic field on the ions when moving in a uniform electric field.

The experimental data indicate that the ratio of the mass to that of the hydrogen atom is the same for the ions from carbon and platinum, and this is very nearly equal to the corresponding quantity determined by J. J. Thomson for the ions from iron. The values are respectively: for platinum, 25.7; for carbon, 27.6; and for iron, about 24.

These numbers show that the positive ions cannot be atoms of the metal or atoms of absorbed hydrogen; neither can they be regarded as identical with the positive electrons found by Thomson in the canal rays.

The emitted ions appear to be quite homogeneous, and experiments carried out between 750° and 1200° indicate that the ratio of charge to mass does not depend on the temperature of the hot substance in any simple way.

The mass ratios in terms of hydrogen are not very different from the molecular weights of nitrogen, carbon monoxide, and oxygen, but there is no evidence that the different substances evolve one or other of these gases when heated. The author, on the other hand, thinks it possible that the positive ionisation is due to a real constituent which is common to the different elements examined. In this case it is necessary to assume that the ions carry a charge which is smaller than the generally recognised fundamental electronic charge. H. M. D.

Determination of the Hydration of Ions by Transference. Experiments in the Presence of a Non-electrolyte. EDWARD W. WASHBURN (*Tech. Quart.*, 1908, 21, 288-320).—Experiments have been made to determine the transference of water which accompanies the migration of ions in aqueous solution by the measurement of transport numbers in presence of a third constituent, or reference substance, which remains stationary during the passage of the current. By referring the changes in concentration of the water and of the electrolyte to this third substance, the amounts of each transferred are calculated, and from these "true" transference numbers have been obtained.

Sucrose, raffinose, and arsenious acid were used as reference substances, the first two substances being estimated polarimetrically, the third by means of an iodine solution. The behaviour of arsenious acid is uncertain, on account of its ready oxidisability. A description of the transport apparatus has been previously given (this vol., ii, 805).

The experimental data show that the electrolysis of solutions of potassium, sodium, or lithium chloride containing one of the above reference substances is attended by an increase in the ratio of the quantity of non-electrolyte to that of water at the anode, and by a corresponding decrease at the cathode. It is shown that this is due chiefly, if not wholly, to a transfer of water from the anode to the cathode. From the measured changes, the relative degrees of hydration of the chlorine, lithium, sodium, and potassium ions have been calculated. On the assumption that the chlorine ion is not hydrated, the minimum hydration values of the cations have been calculated; these correspond with the formulæ: $K(H_2O)_{1.3}$, $Na(H_2O)_{2.9}$, and $Li(H_2O)_{4.7}$. From Buchböck's data for hydrochloric acid solutions, the minimum hydration value for the hydrogen ion corresponds with $H(H_2O)_{6.3}$. Absolute values for the hydration cannot be obtained until the hydration value of some one ion has been determined by some other method.

On account of this transference of water during electrolysis, the transport numbers hitherto obtained for more concentrated solutions of the alkali metal chlorides are erroneous. The true transference numbers are found to vary with the concentration much less than the apparent values obtained by the ordinary Hittorf method.

The true values are also given by the method of moving boundaries, and the author's data are found to agree very well with the results of Denison and Steele obtained by this method.

H. M. D.

Variation of the Degree of Dissociation of Certain Electrolytes with Temperature. ADOLFO CAMPETTI (*Atti R. Accad. Sci. Torino*, 1908, 43, 1071—1094).—The author has determined the conductivities, the temperature-coefficients of conductivity, and the degrees of dissociation of solutions of zinc and magnesium sulphates and of sulphuric acid for temperatures varying from 10° to 90° and for concentrations 3.0—0.001*N*.

The variation of the dissociation with the temperature is, as far as its sign is concerned, in agreement with van't Hoff's law. For an interval of concentration which is not too great, the degree of dissociation is best expressed as a function of the concentration by van't Hoff's formula: $K = \alpha^{3/2}c^{1/2}/(1 - \alpha)$, the expression given by Rudolphi, $K = \alpha^2c^{1/2}/(1 - \alpha)$,* yielding somewhat less concordant results.

Integration of van't Hoff's formula, $q = -RT^2/K \cdot dK/dT$, assuming that the heat of dissociation, q , per gram-molecule is constant for the temperature interval, $T_2 - T_1$, leads to the expression $q(T_1 - T_2)/T_1T_2 = 4.58[\log\{\alpha_2^2/(1 - \alpha_2)\} - \log\{\alpha_1^2/(1 - \alpha_1)\}]$, which is found

be approximately true with magnesium sulphate and with sulphuric acid.

In solutions of magnesium sulphate or zinc sulphate, the undissociated molecules give rise to complex molecules.

T. H. P.

Conductivity and Ionisation of Electrolytes in Aqueous Solutions as Conditioned by Temperature, Dilution, and Hydrolysis. HARRY C. JONES and C. A. JACOBSON (*Amer. Chem. J.*, 1908, 40, 355—410. Compare Abstr., 1905, ii, 794).—So many conflicting results have been published on the question of the influence of temperature on dissociation that the authors were induced to re-investigate the subject. The method employed was that previously described by Jones and West (Abstr., 1905, ii, 794), the temperature at which observations were made also being the same. Twenty-eight inorganic salts and six organic acids have been investigated; the molecular conductivity and percentage of dissociation at 0°, 10°, 25°, and 35° at eight dilutions are tabulated, also the temperature-coefficients in each case. The results are in agreement with the well established facts that (1) the molecular conductivity of electrolytes increases with rise of temperature; (2) the conductivity increases with increase in dilution up to a certain maximum value; (3) dissociation increases with increase in dilution up to the point of complete dissociation. At the somewhat low temperatures of these observations, no maximum in the conductivity curve, as described by Sack, Kohlrausch, and Noyes, was observed. Further, it was found that the conductivity of water increases as a linear function of the temperature, but the molecular conductivity of electrolytes in aqueous solution increases as a parabolic function of the temperature from 0° to 35°. The increase in conductivity of electrolytes causing the departure from a straight line is largely due to their being more or less hydrated; the rise in temperature diminishes the extent of this hydration, allowing the ions to move with less friction, and thus show an increase of conductivity. In support of this view, those salts which are strongly hydrated in solution are found to exhibit a greater increase in conductivity with rise of temperature than the salts that are slightly hydrated. The conclusion is also drawn from the results given that nearly all, if not all, electrolytes are hydrolysed in dilute solutions, and this greatly influences the true dissociation value. When the temperature-coefficients expressed as conductivity units are examined, they are all found to increase as the dilution increases, and for every salt they increase with rise of temperature; but the organic acids, on the other hand, have decreasing temperature-coefficients with increase of temperature. J. V. E.

Basicity of Acids as Determined by their Conductivities. M. R. SCHMIDT (*Amer. Chem. J.*, 1908, 40, 305—313).—After considering the work of Miolati and Mascetti (Abstr., 1901, ii, 381) on the effect of alkalis on the conductivity of solutions of various acids, and the anomalous behaviour of hydrogen fluoride solutions in this connexion (Pellini and Pegoraro, Abstr., 1907, ii, 860), the author gives his results of a study of the conductivity change when potassium

hydroxide is added to moderately strong solutions of phosphoric acid. Working with 0.2*N*, 0.1*N*, 0.04*N*, and 0.02*N* solutions of phosphoric acid, it was found that in the case of 0.2*N* solutions, the minimum occurs when only about 40% of the amount of potassium hydroxide required for one-third neutralisation had been added. In the more dilute solutions, the minimum shifts continually towards the point corresponding with the salt KH_2PO_4 , reaching it with 0.02*N* solutions. As a possible explanation of this, it is suggested that, as more and more potassium hydroxide is added, the H^+ ions of the phosphoric acid are replaced by K^+ ions, which move more slowly, and, as a result, the conductivity of the mixture decreases. But phosphoric acid being far from completely dissociated in strong solutions, when a small quantity of base is added, practically all the hydrogen ions present are neutralised. Immediately, however, more dissociate, and in sufficient quantity to overbalance the decrease in conductivity caused by the substitution of K^+ ions for the H^+ ions, thus causing the conductivity to rise again.

A minimum thus appears before enough alkali has been added to form the primary salt. Furthermore, as the dilution increases, the phosphoric acid becomes more and more completely dissociated according to the formulation $\text{H}_3\text{PO}_4 \rightleftharpoons \text{H}^+ + (\text{H}_2\text{PO}_4)^-$, thus lessening the overbalancing effect of secondary dissociation previously mentioned.

J. V. E.

Comparative Investigations on the Formation of Salts and the Basicity of Acids. GIUSEPPE BRUNI (*Zeitsch. Elektrochem.* 1908, **14**, 729—734).—The molecular conductivity of an acid of which a fraction x is neutralised is $\Lambda = x(l_a + l_s) + (1-x)\alpha(l_a + l_s)$, where l_a , l_s , and l_h are the mobilities of the metallic ion, the anion, and the hydrogen ion respectively, and α is the fraction of the acid which is dissociated. The salt is supposed to be completely dissociated. Assuming that the acid follows Ostwald's dilution law, α can be expressed in terms of known quantities. This gives an expression for the conductivity which reproduces all the peculiarities of the curves obtained by the author (this vol., ii, 935).

T. E.

Certain Phenomena in Gases Subjected to the Action of Spark Discharge or Arc Light. HEINRICH RAUSCH VON TRAUBENBERG (*Physikal. Zeitsch.*, 1908, **9**, 713—726).—Measurements have been made of the electrical conductivity of certain gases during, and after, the passage of electricity through them in the form of a spark discharge and of direct and alternating current arcs. The gases examined were hydrogen, oxygen, acetylene, carbon dioxide, air, coal gas, and carburetted hydrogen. The high conductivity, which is observed during the period of the spark or arc discharge in all cases, persists for several hours after the discharge is stopped in the case of acetylene and coal gas. The phenomenon was investigated more closely in the case of coal gas, and found to depend on the material of the electrodes, and on the nature of the discharge. In

some cases the conductivity, which is unipolar in nature, was found to decrease slowly, whilst in others it increased. No evidence of a saturation current could be obtained. The last part of the paper is devoted to a theoretical discussion of the possible modes of electrical transport through the conducting gases.

H. M. D.

Hysteresis of Certain Iron Compounds. G. BERNDT (*Physikal. Zeitsch.*, 1908, 9, 750—752; *Ber. deut. physikal. Ges.*, 1908, 6, 662—666*).—The magnetic properties of ferric oxide, ferric hydroxide (impure), ferric chloride, and ferrous and ferric sulphates have been examined. The oxide and hydroxide exhibit well-developed hysteresis; in the case of the salts, no such effect could be detected. As the strength of the field increases, the magnetic susceptibility of the oxide diminishes considerably, and that of the hydroxide slightly.

H. M. D.

Magnetic Properties of the Oxygenated Metallic Radicles. P. PASCAL (*Compt. rend.*, 1908, 147, 742—744. Compare this vol., ii, 756, 927).—By measuring the magnetic susceptibilities of their solutions, the author has obtained the following values for the molecular magnetic susceptibilities at 23°: of manganese sulphate, $+1500 \times 10^{-5}$; potassium permanganate, $+25.5 \times 10^{-5}$; chrome alum, $+2 \times 629 \times 10^{-5}$; chromic acid, -0.5×10^{-5} ; vanadium chloride, $+139 \times 10^{-5}$; sodium metavanadate, -30.2×10^{-5} . Comparison of each pair of salts shows that oxidation of the metal results in a diminution in the magnetic susceptibility; the molybdates and ferrates also, unlike the salts of molybdenum and iron, are diamagnetic.

The values for vanadium compounds: VCl_3 , $+139 \times 10^{-5}$; $(\text{V}_2\text{O}_5)\text{SO}_4$, $+2 \times 115 \times 10^{-5}$; vanadium tetrachloride (hydrolysed) ($= \text{V}_2\text{O}_5$), $+66.2 \times 10^{-5}$, and NaVO_3 , -30.2×10^{-5} , indicate that the magnetic properties of the vanadium ion diminish in proportion to the increase in the degree of oxidation; similarly, $\text{U}(\text{SO}_4)_2$ has $+320 \times 10^{-5}$, and $\text{UO}_3(\text{SO}_4)$, -5.57×10^{-5} .

These examples indicate the increasing generality of the law previously enunciated, that when the chemical properties of a metal become masked by its entry into a complex ion or colloid, the magnetic properties are similarly affected and to the same degree. An interesting example of this is afforded by vanadic anhydride, which dissolves in concentrated sulphuric acid to a red solution. The latter on dilution becomes first yellow and then colourless. The anhydride in the red solution has the molecular magnetic susceptibility $-2 \times 85.7 \times 10^{-5}$; in the yellow solution, $-2 \times 31.5 \times 10^{-5}$, and in the colourless solution, $-2 \times 30.2 \times 10^{-5}$. From these values, it is permissible to conclude that the strongly acid solutions contain red sulphovanadic compounds which dissociate on dilution. These complexes doubtless include Berzelius' vanadyl sulphate, $\text{VO}_2(\text{SO}_4)_3$, Ditte's compound, $\text{V}_2\text{O}_5 \cdot 3\text{H}_2\text{SO}_4$, and Gerland's salt, $4\text{SO}_3 \cdot \text{V}_2\text{O}_5 \cdot \text{K}_2\text{O}$.

E. H.

Measurements with Gas Thermometer. ARTHUR L. DAY and J. K. CLEMENT (*Amer. J. Sci.*, 1908, 26, 405—463).—The

* and *Ann. Physik*, 1908, [iv], 27, 712—734.

authors have endeavoured to improve the accuracy of the nitrogen in platinum standard gas thermometer between 0° and 1200° .

Particular attention was paid to uniformly heating the bulb by a series of independently regulated platinum coils whilst the unheated space between bulb and manometer was reduced to a minimum.

The whole furnace being contained in a bomb in which nitrogen was maintained at the same pressure as the thermometer, diffusion and strain on the bulb were obviated, so that much higher gas pressures could be employed than hitherto. The coefficient of expansion of the metal of the bulb was determined with great care.

The melting points of the purest commercially obtainable metals are given as follows: zinc, $418.5^{\circ} \pm 0.1$; silver, $958.3^{\circ} \pm 0.5$; gold, $1059.3^{\circ} \pm 1.0$; copper, $1081.0^{\circ} \pm 0.5$. Exact analyses of the metals are given, but different samples of ostensibly the same metal sometimes differed by 1° in melting point. It is claimed that the determinations of temperature are accurate to within half a degree. R. J. C.

Heat of Fusion, Specific Cohesion, and Molecular Weight at the Melting Point. PAUL WALDEN (*Zeitsch. Elektrochem.*, 1908, 14, 713—724).—For non-associated substances, the ratio L/a^2 is roughly a constant and equal to 3.6 (L = heat of fusion, and a^2 the height to which the liquid rises in a capillary of 1 mm. radius at the melting temperature). The expressions ML/T and Ma^2/T (M = molecular weight, and T absolute melting point) are also roughly constant for non-associated substances. The value of the former is 13.5, and that of the latter, 3.65. For associated substances, the degree of association may be obtained by dividing the value of the expression for any substance into the above constants. The results are in harmony with values obtained by other methods. By combining these results with Trouton's rule and van't Hoff's equations for the depression of freezing point and elevation of boiling point of solutions, it is shown that $E_1/E = 0.64T_1/T$ and $L/L_1 = 0.64T/T_1$, where E_1 , T_1 , and L_1 are the molecular elevation of boiling point, the absolute boiling point, and the heat of evaporation of a liquid, E , T , and L being the corresponding quantities at the freezing point. The application of the equations to some metallic and non-metallic elements and fused salts gives values for the degree of association which agree with what is known on other grounds about the size of their molecules. T. E.

Electrical Heating in Ebullioscopic Determinations and in Fractional Distillation. ERNST BECKMANN (*Zeitsch. physikal. Chem.*, 1908, 64, 506—507. Compare this vol., ii, 663; also Richards and Mathews, this vol., ii, 828).—When, in spite of electrical heating, superheating and bumping occur in ebullioscopic determinations, the difficulty can be got over by passing a current of an indifferent gas through the liquid.

An apparatus is sketched in which electrical heating has been successfully employed for the fractional separation of the chlorides of sulphur. In this way the chloride SCl_2 has been distilled off almost pure at -24° under a pressure of 4 mm. J. C. P.

Quartz Protecting Tubes in Thermal Analysis. P. SCHORN (*Metallurgie*, 1908, 5, 635—637).—Quartz tubes are to be preferred to porcelain for the protection of thermo-couples in the determination of freezing points of alloys, on account of their greater cheapness, and of the less resistance to the passage of heat. They are also less permeable than porcelain to metallic vapours. They are, however, more readily attacked by oxides. They can be used up to nearly 1500°, at which temperature they soften.

C. H. D.

Cryoscopic Investigations on Solutions of Gases in Liquids. PIETRO FALCIOLA (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 324—330).—The author has extended the investigations of Garelli and Falcicola (*Abstr.*, 1904, ii, 312) to the gases, carbon monoxide, nitrogen, hydrogen, oxygen, methane, and nitrous oxide, and to other solvents than those previously employed.

With water or formic acid, carbon monoxide produces a depression of the freezing point, whilst with acetic acid, benzene, and a number of other organic solvents, the freezing point is raised. Similar elevation of the freezing point of certain solvents is also produced by nitrogen, hydrogen, oxygen, and methane, but is not observed with carbon dioxide, hydrogen sulphide, acetylene, or nitrous oxide.

Benzene, the freezing point of which has been raised by means of carbon monoxide, assumes its normal freezing point gradually in the cold, but rapidly on heating. Bromoform, however, through which carbon monoxide or nitrogen has been passed, maintains its elevated freezing point for some days if kept in the dark and out of contact of the air. The phenomenon does not appear to be due to chemical action, but is probably caused by part of the gas adhering to the solvent and separating with the latter in the solid state.

The following table gives the coefficients of absorption of the various gases, calculated from the equation $c = m\Delta/k$.

	H ₂ .	O ₂ .	N ₂ .	CO.	CH ₄ .	N ₂ O.
Water	—	—	—	0.0222	—	—
Formic acid ...	0.0064	0.2057	0.495	0.0600	0.2285	—
Acetic acid.....	0.0041	0.164	0.1153	—	0.0328	0.891
Benzene	—	—	—	—	0.2286	—
Nitrobenzene ...	—	—	—	—	—	0.2926

T. H. P.

An Electrically Heated Oven, with a Liquid Regulator. L. EHLMANN (*Bull. Assoc. chim. Sver. Dist.*, 1908, 26, 272—274).—The apparatus described was constructed for the purpose of maintaining a moderately low temperature in an oven. An ordinary electric lamp is used as the source of heat, and the temperature is regulated by means of a tall cylindrical vessel containing very dilute sulphuric acid. A glass tube carrying a platinum wire is inserted through a cork in a tubulure at the bottom of the cylinder, and a long glass tube carrying a second platinum wire is passed through a cork at the top of the cylinder. This upper tube is made to move freely through the cork, so that the distance between the upper and lower platinum wires may be adjusted as desired. The cylinder is inserted in the circuit feeding

the lamp, and by altering the distance between the platinum wires the degree of heat obtained from the lamp may be regulated as desired. A second form of regulator is also described. It consists of a glass vessel containing the acidified water, and in the latter are placed two copper plates, one fixed and the other movable, the method of using this regulator being similar to that mentioned above. In both cases, means are provided for changing the direction of the current from time to time in order to prevent polarisation. W. P. S.

Thermodynamics of Salts containing Water of Crystallisation. HERMANN SCHOTTKY (*Zeitsch. physikal. Chem.*, 1908, 64, 415—448).—The author applies Nernst's theory (Abstr., 1907, ii, 153) to the case of formation of a higher hydrate from a lower one and water, and deduces from it the relationship $Q - A = T(C - C')$, where Q and A are referred to the combination of 1 molecule of water, C is the molecular specific heat of ice, and C' is the heat capacity of the combined molecule of water of crystallisation. For the purpose of testing this formula, the values of Q and $C - C'$ are obtained from thermochemical data, and A is calculated from the tension of the vapour over the hydrates.

The four cases selected for examination are: (1) $\text{CuSO}_4 + \text{H}_2\text{O} = \text{CuSO}_4 \cdot \text{H}_2\text{O}$; (2) $\text{K}_4\text{FeCy}_6 + 3\text{H}_2\text{O} = \text{K}_4\text{FeCy}_6 \cdot 3\text{H}_2\text{O}$; (3) $\text{BaCl}_2 + \text{H}_2\text{O} = \text{BaCl}_2 \cdot \text{H}_2\text{O}$, and (4) $\text{BaCl}_2 \cdot \text{H}_2\text{O} + \text{H}_2\text{O} = \text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

In order to supplement the few data already available in these cases, the author has determined (1) the specific heats of the hydrates; (2) the heats of hydration; (3) the dissociation pressures. The values obtained differ in some cases from those recorded by earlier investigators, and are employed to show that the formula $Q - A = T(C - C')$ is qualitatively correct in all four cases, and that for (1) and (3) it is capable of quantitative verification within the limits of experimental error. In the other two cases, the quantitative validity of the formula is only approximate.

The difficulty of determining the dissociation pressures of salt hydrates is discussed, and various forms of apparatus suitable for the determination are described. The sluggishness with which the maximum tension is reached is connected with the character of the efflorescing surface.

The theoretical and experimental work of Johnston (this vol., ii, 358) are adversely criticised. J. C. P.

Thermochemical Equivalence, and the Thermochemistry of Nitrogen. JOHN C. THOMLINSON (*Chem. News*, 1908, 98, 226. Compare Abstr., 1907, ii, 153).—In the belief that the numbers given previously may actually represent the thermal changes taking place, the author calculates from the structural formula he gives for nitrogen pentoxide and nitric acid an approximate heat of formation of 12,504 cal. and 45,447 cal. respectively. The experimental values are 13,000 cal. and 41,600 cal. J. V. E.

Apparent Deviations from Mariotte's Law and their Influence on the Measurement of Small Pressures. KARL SCHEEL and WILHELM HEUSE (*Physikal. Zeitsch.*, 1908, 9, 784—789; *Ber. deut. physikal. Ges.*, 1908, 6, 785—793).—The discrepancies

between the results of previous measurements of the compressibility of gases at low pressures have led the authors to examine the influence of a variation in the ratio of the exposed surface to the volume of the containing vessel in the case of air. In order to obtain a very large surface one of the containing vessels was filled with about 20 grams of glass wool. For a variation in pressure from 0.07 to 0.7 mm. of mercury, p_v remained constant in the absence of the glass wool, but when this was present, p_v was found to diminish as the pressure was increased. After drying the glass wool by exhausting the apparatus, and leaving in contact for some time with phosphoric oxide, the variation in p_v could no longer be observed.

The authors conclude that the deviations from Boyle's law which Baly and Ramsay found when air was compressed at low pressures in capillary tubes are due to the presence of a film of condensed water on the surface of the glass containing vessels; when this is removed, the apparent deviations disappear, and the same results are obtained whether the gas is compressed in vessels with a small or with a large amount of exposed surface.

It is pointed out that when the McLeod gauge is used for the measurement of small pressures, all traces of water vapour must be removed from the gas and from the walls of the glass vessel.

H. M. D.

Viscosity and Fluidity. EUGENE C. BINGHAM (*Amer. Chem. J.*, 1908, 40, 277—280. Compare Abstr., 1906, ii, 218).—It is found with pure liquids that the fluidities of unassociated substances are approximately a linear function of the temperature, the relation being expressed by the formula $t_1 = A\phi + K_1$, where t is the temperature absolute, ϕ the fluidity, and A and K_1 are constants. Associated compounds, however, especially the alcohols, depart widely from the linear type, except at high temperatures, when the fluidity curves approximate to a straight line. To represent the increase in temperature required to give the fluidity ϕ on account of the association of the substance, use is made of the expression $t_2 = K_2 - B/\phi$, where B and K are constants. Added together, these two equations become $t = t_1 + t_2 = A\phi - B/\phi + C$, which, when tested with Thorpe and Rodger's data (*Trans.*, 1897, 71, 374) for substances not highly associated, gives results in closer agreement with observed facts than does Slotte's equation. With associated substances, such as acids, alcohols, and water, fairly close agreement is also observed. An approximate equation containing four constants, $t = A\phi - B/(\phi + D) + C$, is also used; it gives with associated substances results in as close agreement with observed values as does the above simpler equation with unassociated substances. Tables comparing the values for hexane and water, calculated by the various equations, are given.

J. V. E.

Anomalies in the Viscosity of Emulsions and of Anisotropic Liquids. EMIL BOSE (*Physikal. Zeitsch.*, 1908, 9, 707—708).—The changes observed in the viscosity of anisaldazine in the neighbourhood of the clearing point (this vol., ii, 258) cannot be used as a proof against the emulsion theory of anisotropic liquids. On the other

hand, Vorländer's experiments (this vol., i, 641) indicate that this theory is untenable.

H. M. D.

Influence of Electrolytes on the Viscosity of Colloidal Liquids. MANFREDI ALBANESE (*Arch. exp. Path. Pharm., Suppl.*, 1908, 16—28).—The viscosity of an aqueous solution of gum arabic is lowered (up to 30%) by the addition of electrolytes, but not by other substances. Solutions of certain other colloids, such as egg-albumin, are not affected by electrolytes, unless they have been dialysed previously, in which case the viscosity is somewhat diminished by adding salt.

G. B.

Capillary Properties of Aqueous Solutions of Fatty Acids. BOHDAN VON SZYSZKOWSKI (*Zeitsch. physikal. Chem.*, 1908, 64, 385—414. Compare this vol., ii, 827).—In contrast with the usually accepted view, the author finds that the heights to which water and fatty acid solutions rise in capillary tubes are almost independent of accidental impurities. For the purest water obtainable, for ordinary distilled water, for conductivity water, and for tap-water, the observed capillary rise is the same to within 0.1 mm. It is also found unnecessary to take special precautions in protecting the capillary tubes from dust; washing out with chromic acid mixture is quite adequate preliminary treatment.

The acids specially examined were *n*- and *iso*-butyric acids, *n*- and *iso*-valeric acids, and *n*- and *iso*-hexoic acids, and the experiments show that the higher an acid stands in the homologous series the greater is its effect in lowering the capillarity of water; the effects of the *n*- and *iso*-acids are nearly equal. In the case of *isobutyric* acid, the variation in the capillarity with concentration is satisfactorily reproduced by the empirical formula $y = 1 - b \log(x/a + 1)$, where *a* and *b* are constants, *x* is the concentration, and *y* is the capillary rise referred to that of water as standard. The other acids examined obey this dilution law only approximately.

In most cases there are considerable differences between the author's measurements and those recorded by Drucker (*Abstr.*, 1905, ii, 680); this is probably to be attributed to the different purity of the acids employed.

Some experiments made on the relative influence of *isovaleric* acid and its barium salt, and on the effect produced by mixtures of the two, support the view that the depression of the capillarity of water is to be attributed to the undissociated part of the acid.

Experiments made with mixtures (1) of *isobutyric* and *isovaleric* acids, and (2) of *isobutyric* and *isohexoic* acids, indicate that the depression of the capillarity of water is an additive property for the fatty acids. In the first case, this fact may be made the basis of an analytical determination of the mixed acids.

J. C. P.

Osmotic Pressure. JOHANNES J. VAN LAAR (*Zeitsch. physikal. Chem.*, 1908, 64, 629—632).—The formula for the osmotic pressure of a perfect solution proposed by Lewis (this vol., ii, 465) is identical with that obtained by the author (*Abstr.*, 1895, ii, 107; 1906, ii,

526) on thermodynamic grounds. The formula obtained in this way is valid for imperfect, as well as for perfect, solutions, and is in accordance with the experimental results of Morse and Frazer (Abstr., 1905, ii, 475; 1906, ii, 600, 601).

C. H. D.

The Vapour Pressure and Osmotic Pressure of a Volatile Solute. HUGH L. CALLENDAR (*Proc. Roy. Soc.*, 1908, A, 81, 336).—If an osmotic membrane is impermeable to the solute, the formulae for the change of vapour pressure with hydrostatic pressure, and for osmotic pressure, are the same for a volatile as for a non-volatile solute. If, on the other hand, the membrane is permeable to the vapour, but not to the liquid phase, the equation depends on the concentration of the constituents in the vapour phase.

C. H. D.

Osmotic Pressure of Sucrose Solutions at 15°. HARMON N. MORSE and B. MEARS (*Amer. Chem. J.*, 1908, 40, 194—213. Compare Abstr., this vol., ii, 671).—In order to ascertain the cause of the osmotic pressure of sucrose solutions at 0° not being in agreement with the gas pressure, as at 20°, measurements at intermediate temperatures have been made. The authors now give results of measurements made at 15°, and from a comparison with those obtained at 0°, 5°, and 10°, previously recorded (*loc. cit.*), they are able to state that sucrose solutions exhibit a temperature-coefficient which is practically identical with the temperature-coefficient of gases.

J. V. E.

Electric Osmosis. JOSEPH C. W. FRAZER and HARMON N. HOLMES (*Amer. Chem. J.*, 1908, 40, 319—325).—A description is given of the apparatus used, and also the results of a preliminary investigation of the relative osmosis of a few salts of nitric acid at the concentration 0.001*N*. The observations recorded for nine different salts appear to support the statement made previously (Abstr., 1906, ii, 600) that the osmosis of the nitrates of alkalis and alkaline earths varies inversely as the velocity of the cation divided by its valency, but the osmosis does not seem to be proportional to the specific resistance of the solutions.

It is suggested that some connexion may exist between this phenomenon and the results obtained by Kohlrausch (Abstr., 1903, ii, 403) and others working on the hydration of ions.

J. V. E.

Improvement in Cells for the Measurement of Osmotic Pressure. HARMON N. MORSE and B. MEARS (*Amer. Chem. J.*, 1908, 40, 266—277).—A detailed account is given of the weak points of the cell previously used and described (Abstr., 1905, ii, 575); the paper deals, however, in general with the form of cells and the method of closing them for the measurement of osmotic pressure. A new form of cell is described, which is entirely free from the objectionable features of the older one. The upper end of it is glazed inside and out, and has an accurately ground conical aperture at the top for the reception of a brass, cone-shaped collar, which is covered with a caoutchouc cap carrying the manometer

tube. The closing of this cell is rapidly effected by screwing a brass nut down on to the collar. For details of construction and manipulation, the original must be consulted. J. V. E.

Improved Manometers for Measurement of Osmotic Pressure. HARMON N. MORSE and B. F. LOVELACE (*Amer. Chem. J.*, 1908, 40, 325—337).—An apparatus is described by means of which manometers may be compared with others, or with the standard, up to pressures of three hundred atmospheres. Corrections for meniscus, and a method of detecting a temporary or a permanent stretch of the manometer, are also given, as well as a detailed description of the method used by the authors for filling and closing their manometers. J. V. E.

The System Water, and Sodium, Barium, and Copper Chlorides. FRANS A. H. SCHREINEMAKERS and W. C. DE BAAT (*Chem. Weekblad*, 1908, 5, 801—806).—The authors represent the quaternary system water, sodium chloride, barium chloride, and copper chloride by means of a regular tetrahedron, the four angles giving the four components. The method is an extension of that employed to represent a ternary system by means of an equilateral triangle. A. J. W.

Velocity of Reactions in Gases Moving through Heated Vessels, and the Effect of Convection and Diffusion. IRVING LANGMUIR (*J. Amer. Chem. Soc.*, 1908, 30, 1742—1754).—In studying the changes which take place in mixtures of gases while passing through hot tubes, the velocity coefficient of the reaction is usually calculated by means of the formula derived for reactions occurring in stationary gases. Bodenstein and Wolgast (this vol., ii, 162) have pointed out that this method is inaccurate, since allowance is not made for the mixing of the gases in the tube by diffusion or convection, and they have presented a formula which holds when the mixing of the gases can be considered complete.

In the present paper, these two formulæ and the magnitude of the error arising from their application in various cases are considered. New formulæ are developed, from which the velocity coefficient may be calculated in cases in which neither of the formulæ mentioned holds good, but in which, in the mixing of gases, diffusion plays a more important part than convection. E. G.

Relation between the Velocity of Reaction and the Velocity of Stirring in Non-homogeneous Systems. K. JABŁCZYŃSKI (*Bull. Acad. Sci. Cracow*, 1908, 620—631).—The rate of dissolution of a solid in a liquid increases with stirring. The connexion between the velocity constant k and the velocity of the stirrer n is given by the empirical equation $k_1/k_2 = (n_1/n_2)^x$, where x is a constant, the value of which varies with different apparatus, and even with the same apparatus if taken apart and put together again. This probably accounts for the different values of x obtained by different investigators (Bruner and Tolloczko, *Abstr.*, 1903, ii, 470; Bruner, *Abstr.*, 1904, ii, 315; Sackur,

Abstr., 1906, ii, 261). The evaluation of α is important, since it gives some information as to how far a reaction in a non-homogeneous system is dependent on diffusion (Noyes and Whitney, Abstr., 1897, ii, 479; Nernst, Abstr., 1904, ii, 315). In the same apparatus, so arranged that each experiment can be completed without any disturbance of the parts of the apparatus, the author has examined, at 25°, the solution of a marble plate in hydrochloric acid, the decomposition of hydrogen peroxide by platinised platinum foil, and the solution of a fused zinc plate in hydrochloric acid. The average values of α are 0.93, 0.55, and 0.28 respectively. Hence the author deduces that, for the dissolution of a solid with a perfectly smooth surface, the velocity constant of the reaction is simply proportional to the velocity of stirring (as measured by the revolutions of the stirrer); if the surface of the solid is not smooth, the increase of the velocity constant is slower than the increase of the velocity of stirring, and the more so the rougher the surface.

C. S.

Saponification of Glycerides during Ester Exchanges in Homogeneous Systems. ROBERT KHEMANN (*J. pr. Chem.*, 1908, [ii], 78, 364—367); MILAN J. STRITAR and RICHARD FANTO (*ibid.*, 408). —Polemical (compare Abstr., 1907, i, 464; this vol., i, 120, 499; ii, 677).

G. Y.

Oxidations of Biological Importance. I. HANS EULER and IVAN BOLIN (*Zeitsch. physiol. Chem.*, 1908, 57, 80—98).—Careful experiments have been made on the oxidation of quinol by oxygen in the presence of manganous salts and alkali. It is shown that the velocity of the reaction is almost proportional to the pressure of the oxygen, and that the temperature-coefficient is very small between 0° and 40°. The solutions used were either neutral or slightly acid to phenolphthalein. To obtain such solutions, it is necessary to add dilute alkali, as quinol is decidedly acidic. The effect of the manganese (added as acetate) and of the sodium concentrations has been studied. As the whole reaction cannot be represented by means of the usual unimolecular formula, owing to the fact that part of the quinol is removed in the form of insoluble quinhedrone, the velocity has been determined by measuring the times required in the different experiments for absorbing a given volume (3 or 5 c.c.) of oxygen. The results show that in neutral or slightly acid solutions, manganese salts accelerate the oxidation, and that if the amount of sodium hydroxide added is the same, the rate of oxidation increases less rapidly than the manganese concentration. On the other hand, if the amount of manganese salt is constant, the velocity increases much more rapidly than the amount of alkali added.

The relationship between the effects of the manganese salt and the alkali is compared with that of an enzyme and co-enzyme.

Similar experiments have been made with "laccase" and manganese salts. It is shown that the laccase as prepared by Bertrand's method is slightly acid, and that it has no action on a quinol solution in the absence of manganese salts. It is obvious that the action of the laccase cannot be due to its alkaline nature, as suggested by Dony-

Hénault. Even the addition of 10 c.c. of 0.0001*N* hydrochloric acid to lucerne laccase does not affect the activity of the laccase in the presence of manganous salts.

It is further shown that the active principle of the laccase is not a substance of the enzyme type, as the preparations of laccase can be boiled for three minutes without destroying their activity in the least.

It is shown that salts of hydroxy-acids, for example, rochelle salt, sodium citrate, calcium gluconate, and sodium mucate, accelerate the oxidation of quinol in a marked manner when manganese salts are present, and it is suggested that laccase owes its activity to the presence of such salts.

J. J. S.

Some Oxydasic Phenomena Produced by Colloidal Ferrous Ferrocyanide. JULES WOLFF (*Compt. rend.*, 1908, 147, 745—747. Compare this vol., i, 137, 490; ii, 573).—In a feebly alkaline solution, colloidal ferrous ferrocyanide acts as an oxydase towards quinol. When a saturated solution of quinol is treated with ammonia to the extent of 1 part in 25,000, and colloidal ferrocyanide to the extent of 1 part of iron in 100,000, abundant crystals of quinhydrone are formed after twelve to fifteen minutes, oxidation being accelerated by shaking. The reaction is still observed with one-twenty-fifth as much iron if the quantity of alkali is increased to 1 part in 2000. Solutions of quinol treated with alkali alone become brown, but rarely deposit crystals, the colloidal iron alone has no action. If too much alkali (1%) is added, the solution is rapidly oxidised by the air, and no crystals are formed. These phenomena can be explained by the alkali facilitating the oxidation of the phenol, and consequently the action of the ferrocyanide, which thus acts as an oxydase. This view is supported by the facts that if the alkali is added some time before the colloidal iron, quinhydrone is no longer obtained, and that, other things being equal, the intensity of the reaction is proportional to the amount of alkali present (within the limits described above). From the latter observation, it would be expected that the alkali-earth oxides and hydrogen carbonates, and feebly alkaline salts, such as disodium hydrogen phosphate, would exercise a much less energetic action. This is actually found to be the case. The important part played by the alkali suggests that the oxidising action on quinol of certain manganese salts with weak acids discovered by Bertrand (*Abstr.*, 1897, ii, 493) is due partly to their alkalinity, all of them being alkaline to methyl-orange. The author finds that the addition to these solutions of a quantity of iron in the form of colloidal ferrocyanide, one-hundredth as great as that of manganese present, suffices to double the rate of oxidation of quinol, and that neutral aqueous manganese sulphate solutions, which are ordinarily almost inactive, increase their activity considerably when treated with traces of pyridine (which does not precipitate manganese).

E. H.

Catalysis. ~~See~~ Catalytic Reactions induced by Enzymes. SALOMON F. ACREE (*Amer. Chem. Soc.*, 1908, 30, 1755—1760).—Attention is drawn to the work of Hudson (this vol., i, 605, 856)

on the inversion of sucrose by invertase, and it is shown that it harmonises completely with the view already advanced by others, that the addition of an enzyme to a reacting system brings about a catalytic reaction, owing to the union of the enzyme with some constituent or "substratum" of the reacting system to form a new product which gives the same end-products as the substratum itself. It is pointed out that this theory of catalysis through the formation of reactive intermediate compounds renders the assumption of a vital energy for such reactions unnecessary.

E. G.

Molecular State of Molten Salts. RICHARD LORENZ and FELIX KAUFLEDER [in part, A. LIEBMANN] (*Ber.*, 1908, 41, 3727—3738).—The molecular weights of lead chloride, potassium nitrate, sodium nitrate, and silver chloride in the fused state at various temperatures between 300° and 600° have been determined by measuring the surface tension of the molten salt. The mean values of K obtained for potassium nitrate and sodium nitrate, namely, 0.7 and 0.25 respectively, are in fair agreement with those recorded by Bottomley (*Trans.*, 1903, 83, 1421). The constants found for silver chloride and lead chloride were respectively 0.8 and 0.9. Since these values are all less than 2.12, the constant for liquids having a normal mol.-wt., the conclusion is drawn that the salts investigated exist in the fused state in a highly associated condition and that the ions are of a complex nature.

W. H. G.

Liquid Crystals. OTTO LEHMANN (*Ber.*, 1908, 41, 3774—3783).—Mainly a historical review of the author's investigations on the subject of liquid crystals.

The view recently put forward by Vorländer (this vol., i, 641), that pseudo-isotropic, crystalline liquids are to be regarded as uniaxial crystals, is combated. It is possible for a biaxial mass to become pseudo-isotropic, that is, apparently uniaxial.

W. H. G.

The Liquid-crystalline State as a General Property of Matter. P. P. VON WEIMARN (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 166—168*).—Having shown in previous papers (this vol., ii, 90, 263) that the colloidal-amorphous state of matter is a modification of the crystalline, the author has now studied certain liquid crystals, such as *p*-azoxyphenetole, both microscopically and ultramicroscopically, and states his views of the nature of these crystals as follows. A liquid crystal is a system of chemically or physically associated, solid, crystalline particles, between which vectorial forces of small tension act. The small magnitude of the tension in the case of chemical association is conditioned by the considerable energy expended in building up the complicated molecule in the case of physical association by the expenditure of energy requisite for the association of a number of elementary crystalline particles to one particle.

G. S.

Theory of Colloids. EDUARD JORDIS (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 153—166).—A summary of the author's views on the nature of colloidal solutions (compare *Abstr.*, 1907, ii, 344; this vol., ii, 675).

* and *J. Russ. Phys. Chem. Soc.*, 1908, 40, 1323—1327.

The theory developed is in many respects identical with that of Duclaux (summarised in *J. Chim. Phys.*, 1907, 5, 29), and many of Duclaux's results are discussed in detail. Pure hydrosols are not stable; the stability is determined by small amounts of other substances, usually electrolytes, which act as "sol-formers." These substances are in chemical combination with the dissolved colloids, and when they are completely removed, the hydrosol coagulates.

G. S.

Electrical Nature of Colloidal Solutions. NICOLA PAPPADÀ (*Gazzetta*, 1908, 38, ii, 474—480. Compare Abstr., 1907, ii, 754).—The author criticises Duclaux's hypothesis concerning the electrical nature of colloids (compare Abstr., 1904, ii, 162, 243, 325; 1905, ii, 432, 511; 1906, ii, 660, 677; this vol., ii, 760).

T. H. P.

Influence of Colloids on the Absorption of Gases, especially of Carbon Dioxide in Water. ALEXANDER FINDLAY [with W. H. HARRY] (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 169—170).—The absorption of carbon dioxide by blood is not well understood, and it is suggested, in agreement with Wolfgang Ostwald (this vol., ii, 509), that, besides ordinary chemical combination, adsorption on the surface of the colloidal particles may be of importance in this connexion. As the first step in an investigation of the effect of colloids on the solubility of gases, the solubility of carbon dioxide in colloidal solutions of ferric hydroxide, gelatin, arsenic sulphide, silicic acid, albumin, dextrin, soluble starch, and glycogen has been measured at 25°. The first two colloids increase the solubility, the three last mentioned diminish it somewhat, and the other three are without effect.

G. S.

Colloidal Silver (Silver-gel) in Photographic Layers. LÜPP-CHRAMER (*Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 170—174).—For the direct production of positives as employed in ferrotype photography, it is necessary that the silver in the parts which are most strongly lighted should be obtained in a more or less white form. This can now be effected with dry collodion plates covered with colloidal substances, such as tannin or gum.

In order to study the deposition of silver in different forms, silver bromide plates have been exposed in a Chapman Jones "plate tester," which allows of graduated illumination. When a plate thus exposed is developed with ferrous oxalate, the silver varies in appearance from greyish-white, on the most highly exposed parts, to greyish-black, on the parts almost in shade, and the difference persists after fixing. The white silver has a much smaller adsorptive power than the black form, and this difference in properties is employed to account for certain phenomena occurring in photographic processes.

G. S.

Freezing of Hydrosols. O. BOBERTAG, KARL FEIST, and H. W. FISCHER (*Ber.*, 1908, 41, 3675—3679).—The paper gives a review of the behaviour of colloidal solutions at temperatures below the freezing point of the solvent. By rapid cooling to -70° , the metal is separated

from Bredig's colloidal suspension of platinum. By freezing a colloidal solution of arsenic trisulphide, Winter (Abstr., 1905, ii, 245) obtained the solid red modification, the authors cool a very dilute solution to -70° , and, after thawing, recover the yellow colloidal solution, which, however, has become very turbid. Colloidal ferric hydroxide, cooled to -70° and thawed, remains clear, but shows a distinct augmentation of the Tyndall phenomenon. Aluminium acetate behaves somewhat similarly. The colloidal silver preparations, protargol, collargol, and lysargin, in the presence of albumin are dark coloured, very turbid, and optically non-homogeneous; by freezing, the silver collects into small lumps, which are distributed irregularly through the clear ice; on thawing, the solution is recovered apparently unchanged.

In connexion with organic colloids, certain dyes and substances of animal and vegetable origin have been examined. Rhodamine, chrysoidine, eosin, and safranine form true solutions; Nile-blue, acid-violet, methyl-violet, and magenta behave as semi-colloids, and night-blue, alkali-blue, Congo-red, and benzopurpurin as pronounced colloids (compare Freundlich and Neumann, this vol., ii, 820; Höber and Chassin, *ibid.*, ii, 875).

On cooling a 10% solution of tannin, a considerable deposition is observed just above the freezing point; on thawing the frozen mass, the tannin partly dissolves, the remainder going into solution by slight warming. Hæmoglobin (Merck), after being cooled to -10° , -70° , and -180° , shows no apparent change. Solutions of gum or starch, cooled to the same temperatures and thawed, are quite turbid, but clarify by keeping. Dried albumin in solution, cooled to -70° to -180° and thawed, gives a turbid solution, which does not again clarify entirely. Solutions of gelatin, caragheen moss, agar-agar, and soap behave in a similar manner on freezing. The water is mostly frozen out from the jelly, so that the first portion of the liquid obtained by thawing is practically free from dissolved substances. After complete thawing, the product is decidedly non-homogeneous, consisting of a mobile liquid and a clotted jelly.

Attempting to explain the varying behaviour of the preceding colloidal solutions on freezing, the authors suggest that the freezing causes the colloidal particles to become aggregated, and that these aggregations may be partly or entirely destroyed by thawing, with the result that a precipitation or a solution is obtained.

C. S.

Study of Colouring Matters in Solution. LOUIS PELET-JOLIVET and A. WILD (*Compt. rend.*, 1908, 147, 683—685; *Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 174—177; *Bull. Soc. chim.*, 1908, [iv], 3, 1087—1094).—From a study of the electrical conductivity and the ultramicroscopic state of solutions of various dyes (magenta, methylene-blue, crystal-violet, safranine, crystal-ponceau, naphthol-yellow S, Congo-red, etc.), the conclusion is drawn that dyes are dissociated in aqueous solution into two ions, and that some of them assume the colloidal condition. Addition of electrolytes favours the passage into the colloidal state. The authors' results do not entirely agree with those of Teague and Buxton (Abstr., 1907, ii, 933) and of Freundlich and Neumann (this vol., ii, 820).

J. C. C.

Fixation of Different Derivatives of the Same Colouring Matter and Explanation of Dyeing. LOUIS PELET-JOLIVET and N. ANDERSEN (*Compt. rend.*, 1908, 147, 808—810.* Compare preceding abstract).—Wool was placed in solutions containing equivalent quantities of derivatives of certain basic and acidic colouring matters. Other conditions being the same, the hydrochloride of safranine gave a paler tint than the sulphate, and the phosphate a darker tint. The hydroxide gave a tint intermediate between the sulphate and phosphate. The sodium salt of crystal-ponceau produced lighter tints than the magnesium salt; the aluminium salt gave darker tints, whilst the free acid was intermediate between the two latter salts in colouring power.

These experiments confirm the theory of Freundlich and Loser (*Abstr.*, 1907, ii, 155, 534) of the fixation of basic and acidic dyes. The wool is supposed to become negatively charged when placed in water; when treated with a solution of the salt of a basic dye, adsorption of the positively charged organic ion occurs, whilst in the case of the salt of an acidic dye, the positive inorganic ion is adsorbed, causing the fabric to assume a positive charge, which, being smaller than the negative charge produced by an equivalent quantity of a negative dye, accounts for the well-known fact that wool and charcoal fix basic dyes more readily than acidic ones. This accords, moreover, with the effects produced by adding electrolytes to the bath, since positive multivalent ions or hydrogen ions should favour the fixation of the acidic dyes, whilst negative ions should hinder the process. A rational explanation is thus afforded of the action of mordants and of the tanning process.

W. O. W.

Passive State of Metals. Review of the Literature and Theories and Some Experiments on Cobalt, Iron, and Nickel. HORACE G. BYERS (*J. Amer. Chem. Soc.*, 1908, 30, 1718—1742).—A historical and theoretical discussion is given of the work done hitherto on the passivity of metals. Experiments are described which have been carried out with cobalt, iron, and nickel. A plate of the metal connected with a copper conducting wire was suspended as anode in a porous cup, which was placed in a porcelain beaker. Both beaker and cup were nearly filled with solutions of the various electrolytes. The cathodes, two platinum plates of the same size as the anode, were suspended in the beaker parallel to, and at equal distances from, the anode. The amount of the anode dissolved during the passage of the current was measured by its loss of weight.

The results obtained were so varied and conflicting as to render a brief summary impossible. Iron, nickel, and cobalt cannot be rendered passive by the hydrogen acids or their salts. Passivity may take place in electrolytes containing oxygen, and depends on the current density, the temperature, the electrolyte, and the treatment of the metal. The metals may be either passive or active under exactly the same current conditions so long as the current density is below a critical value.

The phenomena of passivity have not yet received any satisfactory explanation. Faraday's view, connecting passivity with the formation of a coating of oxide on the metal, is untenable. The hypotheses of

* and *Zeitsch. Chem. Ind. Kolloide*, 1908, 3, 206—210.

Finkelstein and Müller, according to which the passive state is occasioned by changes in the valency of the metal, are unsatisfactory. Many of the results now obtained cannot be reconciled with Fredenhagen's explanation, which attributes passivity to anodic oxygen or an oxygen alloy.

E. G.

Determination of the Atomic Weight of the Simple Ponderable Substance, Pantogen. GUSTAVE D. HINRICHS (*Compt. rend.*, 1908, 147, 797—800. Compare this vol., ii, 573, 574).—The author assumes the existence of a simple fundamental material of atomic weight $1/128$, and thence proceeds to theoretical deductions as to the weight and geometrical forms of the atoms of hydrogen, helium, nitrogen, oxygen, and fluorine. The paper includes a diagrammatic representation of the atoms of these elements, and of their physical properties in the neighbourhood of the absolute zero.

H. O. W.

Atomic Weight of Radium and Other Elementary Substances. HENRY WILDE (*Phil. Mag.*, 1908, [vi], 16, 824—830. Compare Abstr., 1907, ii, 149; this vol., ii, 141).—The author still maintains that the true atomic weight of radium is 184. A periodic table is given, based on the numerical relationships previously described.

H. M. D.

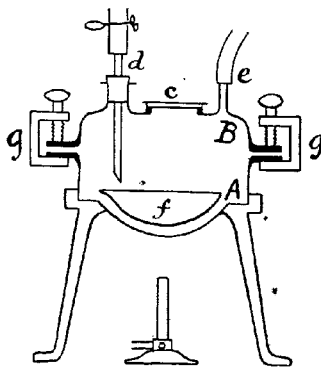
New Form of Test-tube Holder. H. STOLTZENBERG (*Zeitsch. angew. Chem.*, 1908, 21, 2272).—The advantage of the new form of wire holder described lies in the fact that, not only may the tube or other object held be readily released, but the force by which it is held may be increased by the power of the hand. It thus becomes possible to lift fairly heavy vessels and to shake them without fear of their falling.

J. V. E.

Apparatus for Evaporating in a Dish under Reduced Pressure. EDUARD DONATH (*Chem. Zeit.*, 1908, 32, 1107).—The accompanying figure depicts the apparatus described by the author. It consists of two parts, A and B, both being made of stout copper having thick, broad flanges so as to form an air-tight chamber when they are screwed down on to an asbestos washer by screws, g. Into the lower part, A, the evaporating basin, f, is placed upon asbestos fibre.

The upper part, B, is perforated in three places, c, d, and e; c is covered by a thick plate of transparent mica, the liquid to be evaporated is introduced through d, and e is connected to an exhaust pump.

J. V. E.



Circulating Pump. H. STOLTZENBERG (*Zeitsch. angew. Chem.*, 1908, 21, 2271—2272).—A new form of small centrifugal pump is described as being an efficient circulator of liquid from a thermostat through condensers, jacketed polarimeter tubes, etc. The power required to drive the pump is very small, and when rotating at 1200 revolutions per min. it is able to lift water to a height of ten metres.

J. V. E.

Inorganic Chemistry.

[Production of Hydrogen Peroxide from Persulphuric Acid.] CONSORTIUM FÜR ELEKTROCHEMISCHE INDUSTRIE (D.R.P. 199958).—When carefully-purified solutions of persulphuric acid are treated with sulphuric acid (D 1.4) at 50—80°, the former rapidly yields hydrogen peroxide with a loss of only 3 or 5% of oxygen. This result is, however, only obtained in the absence of all impurities likely to decompose hydrogen peroxide catalytically (compare *Trans.*, 1904, 85, 1526—1533).

G. T. M.

Generation of Oxygen in a Kipp's Apparatus. LUDWIG WOLTER (*Chem. Zeit.*, 1908, 32, 1066).—The extremely vigorous evolution of oxygen from alkali peroxides when treated with water or dilute acids has been sufficiently modified by admixture with indifferent substances to allow of the generation of the gas in a Kipp's or other gas generation apparatus by this method.

The best results have been obtained with a substance prepared by adding a mixture of 100 parts of sodium peroxide and 25 parts of magnesium oxide to 100 parts of molten potassium nitrate. This, when solidified and broken into pieces of a suitable size, is placed in a Kipp's apparatus and treated with dilute hydrogen chloride. The substance may be kept for some time ready for use by dipping the lumps into molten paraffin wax so as to protect them from the atmosphere.

J. V. E.

Dynamic Allotropy of Sulphur. I. HUGO R. KRUYT (*Zeitsch. physikal. Chem.*, 1908, 64, 513—561).—The investigations of Smith and his collaborators (*Abstr.*, 1903, ii, 139, 284; 1905, ii, 382, 580; 1906, ii, 157; 1907, ii, 20; this vol., ii, 32) have led to results which accord best with the theory that S_A and S_μ are partly miscible in the liquid state, but that the equilibrium curve lies entirely outside the heterogeneous region. Smith's conclusion, that the equilibrium curve cuts the curve of miscibility, is untenable. A bibliography and review of previous literature on the subject are given.

The melting point of rhombic sulphur was determined by immersing capillary tubes containing the powdered sulphur in boiling calcium chloride baths at different temperatures, and was finally fixed at

112.4°. Similar sulphur, heated for an hour at 90° in ammonia to produce equilibrium, melted at 110.9°. The unstable "natural melting point" of rhombic sulphur was then found graphically to be 110.5°, and the melting point of pure rhombic sulphur 112.8°. From 111° onwards, the melting-point curve must be parallel to the concentration axis.

The composition of the vapour in contact with liquid sulphur at different temperatures was measured by blowing a current of air through the molten sulphur at constant temperature, and condensing the current of vapour on the surface of water at 0°. Ammonia gas was introduced as a catalyst, this being subsequently removed by the current of air, the sulphur dioxide formed by partial oxidation then serving to preserve the composition of the vapour unchanged during cooling. The condensed sulphur was analysed by extraction with carbon disulphide. The vapour and liquid curves intersect at 324° and 24.7% S_μ .

The vapour-pressure curve of the system S_λ — S_μ presents either a maximum or a minimum, but it is not yet possible to determine which. This point is to be further investigated. This curve must cut the curve of equilibrium.

C. H. D.

The Boiling Point of Sulphur on the Constant Pressure Air Thermometer. NICHOLAS EUMORFOPOULOS (*Proc. Roy. Soc.*, 1908, *A*, 81, 339—362).—A detailed account of measurements with a Callendar air thermometer of Jena glass. The various sources of experimental error were very fully investigated, the most important being the changes of volume of the glass bulbs. The mean value of the boiling point of sulphur obtained is 443.58°. C. H. D.

The Boiling Point of Sulphur. HUGH L. CALLENDAR (*Proc. Roy. Soc.*, 1908, *A*, 81, 363—366. Compare preceding abstract).—The changes of volume in glass render it an unsuitable material for accurate thermometry. The value for the boiling point of sulphur obtained by Eumorfopoulos is in close agreement with those obtained by other methods, the chief source of error being now the uncertainty as to the true coefficient of expansion of mercury. C. H. D.

Preparation of Hydrazine. FRITZ RASCHIG (D.R.-P. 198307).—When excess of strong aqueous ammonia reacts with a solution of sodium hypochlorite in the presence of an organic substance to increase the viscosity of the solution, a good yield of hydrazine is obtained. One litre of sodium hypochlorite and 12 c.c. of a solution containing 5% of joiners' glue are added to 3 litres of concentrated aqueous ammonia, the solution heated to drive off ammonia, and concentrated to the crystallising point, when 80—90 grams of hydrazine sulphate are obtained on the addition of sulphuric acid. The product is purified by crystallisation. G. T. M.

Hydroxylamine. I. ERICH EBLER and E. SCHOTT (*J. pr. Chem.*, 1908, [ii], 78, 289—342).—The authors review the various formulæ

which have been proposed for hydroxylamine, and assemble the evidence in favour of the view that it is a tautomeric substance, having in alkaline solution the constitution $\text{NH}_2\cdot\text{OH}$, and acting as an acid, but in acid solution behaving as an oxonium base, NH_3^+O , forming salts of the type $\text{NH}_3^+\text{O} \begin{smallmatrix} \text{H} \\ \diagup \\ \text{X} \end{smallmatrix}$. This view of the nature of hydroxylamine is applied to the explanation of its properties and of the reactions which it undergoes.

Anhydrous hydroxylamine is readily prepared by dissolving 4–5 grams of the 75% distillate obtained by Uhlenhuth's method (Abstr., 1900, ii, 475) in 100–400 c.c. of absolute alcohol, and cooling the solution to about -18° . Hydroxylamine is deposited from the stronger solutions in white leaflets, and from the more dilute solutions slowly in needles.

Lobry de Bruyn found (Abstr., 1892, 1391) that hydroxylamine reacts with zinc, forming zinc oxide and ammonia. It is now shown that the intermediate product of this reaction is a zinc salt, $\text{Zn}(\text{O}\cdot\text{NH}_2)_2$, and not an additive compound, $\text{Zn}\cdot\text{O}\cdot\text{NH}_2$. A similar calcium salt has been prepared, and indications obtained of the existence of an extremely unstable ferric salt. It is proposed to apply the term hydroxylamites to the salts $\text{NH}_2\cdot\text{OM}'$, $(\text{NH}_2\cdot\text{O})_2\text{M}'$, etc.

Calcium hydroxylamite, $\text{Ca}(\text{O}\cdot\text{NH}_2)_2$, is formed with evolution of gas when calcium filings are treated with anhydrous hydroxylamine at 50° . Ammonia is evolved only if heat is developed, which takes place if water is present. The white salt, which separates towards the end of the reaction, is extremely explosive, being much more dangerous than anhydrous hydroxylamine; when washed with absolute alcohol and dried in a vacuum over sulphuric acid, it is obtained as a white, amorphous powder, which detonates at 180° , and is hydrolysed by water, slowly by moist air, forming calcium hydroxide and hydroxylamine. It is considered that Hofmann and Kohlshütter's calcium salt of hydroxylamine (Abstr., 1898, ii, 380) is a mixture of calcium hydroxylamite and calcium hydroxide.

Zinc hydroxylamite, $\text{Zn}(\text{O}\cdot\text{NH}_2)_2\cdot 3\text{NH}_3\text{O}$, prepared by the action of anhydrous hydroxylamine on finely-divided pure zinc and treatment of the product with absolute alcohol, is obtained in glistening crystals, loses hydroxylamine, and leaves a residue of zinc oxide when gradually heated, detonates, evolving ammonia, when quickly heated, and is readily hydrolysed by moisture, forming zinc hydroxide.

Haber observed (Abstr., 1898, ii, 23) that hydroxylamine oxidises ferrous salts in ammoniacal or sodium carbonate solution, and stated that the hydroxylamine is itself converted into ammonia. It is now found that under these conditions nitrogen and nitrous oxide are first evolved, ammonia appearing only towards the end of the reaction. The study of the reaction is complicated by the fact that hydroxylamine is decomposed catalytically by ferric hydroxide in neutral or alkaline solution, yielding the same three products. The ferric hydroxide residue is scarlet, becomes brownish-red when heated, does not decolorise permanganate or evolve chlorine from hydrochloric acid, and thus resembles the ferric hydroxide residue obtained from the catalytic

decomposition of hydrogen peroxide. If hydroxylamine is treated with ferric hydroxide in absolute alcoholic solution at about -18° , a brown precipitate is formed, which, after being washed with absolute alcohol, decomposes on treatment with water, forming nitrogen, nitrous oxide, and ammonia, and must therefore contain *ferric hydroxylamite*.

Hydroxylamine silicofluoride, $(\text{NH}_3\text{O})_2\text{H}_2\text{SiF}_6 \cdot 2\text{H}_2\text{O}$, prepared by adding 16% aqueous hydrofluosilicic acid to a 25% aqueous solution of hydroxylamine and allowing the mixture to evaporate, crystallises from boiling methyl alcohol in scales, and is almost insoluble in alcohol, but readily dissolves in water.

Hydroxylamine titanofluoride, $(\text{NH}_3\text{O})_2\text{H}_2\text{TiF}_6$, prepared by adding 20% hydroxylamine to a cooled solution of hydrofluotitanic acid and evaporating the solution in a vacuum, separates from methyl alcohol in white crystals. With quadrivalent titanium, hydroxylamine gives the same yellow coloration, caused by the formation of a higher oxide of titanium, as is produced by hydrogen peroxide.

G. Y.

The Interaction of Nitrous Gases and Oxygen with Water.
FRITZ FOKKSTER and M. KOCH (*Zeitsch. angew. Chem.*, 1908, 21, 2209—2219. Compare this vol., ii, 941).—In continuation of this investigation (*loc. cit.*), the authors have studied the behaviour towards water of nitric peroxide when greatly diluted with air. The results, which are tabulated, show the proportion of nitric peroxide converted into nitric acid to be greater when the concentration of this gas in the gaseous mixture is greater; it is, however, not directly proportional, being considerably less than would be expected. This may be explained by the fact that with large excess of air the nitrous acid represented in the equation $\text{H}_2\text{O} + 2\text{NO}_2 \rightleftharpoons \text{HNO}_3 + \text{HNO}_2$, being less stable in solution, allows of a more rapid absorption of fresh nitric peroxide than when the excess of air is less. It is also shown that with diluted nitric oxide the increased concentration of nitric acid is not due simply to absorption of more oxides of nitrogen, but, in part, to the nitric acid formed previously leaving the solution with water as $\text{HNO}_3 \cdot \text{H}_2\text{O}$. The limiting concentration at the ordinary temperature at which this takes place is indicated by the following: with 1% NO_2 , about 46% HNO_3 ; with 2% NO_2 , about 51.8% HNO_3 , and with 5% NO_2 , more than 55.6%.

From a study of the influence of a large excess of air on the behaviour of the nitrous acid produced in the primary change, it is shown that it completely decomposes, with time, into nitric acid and nitric oxide. Excess of oxygen, when not at the same time under a smaller partial pressure, rapidly oxidises this nitric oxide to nitric peroxide, but, in the case of a large excess of air, there is insufficient time for the nitric oxide that is carried forward from the water solution to be completely oxidised to the peroxide. The quantity of nitric oxide escaping oxidation was ascertained by substituting for the water, dilute alkali hydroxide solution as absorbent, and the quantity of nitrite produced is shown to be greater when the dilution by air is greater.

Nitric peroxide, even when largely diluted by air, is rapidly

oxidised by ozone to nitrogen pentoxide, and when such a gaseous mixture is passed into water, or aqueous nitric acid, an acid solution containing more than 80% HNO_3 may readily be obtained.

J. V. E.

Formation of Colloidal Phosphorus. ALFRED LOTTERMOSER (*J. pr. Chem.*, 1908, [ii], 78, 367—368).—When exposed to diffused daylight in a closed vessel, a clear, colourless solution of white phosphorus in contact with solid white phosphorus gradually becomes opalescent and orange-yellow by reflected, but clear and yellowish-red by transmitted, light. The hydrosol thus formed is unstable, and, after some time, deposits a scarlet crust of red phosphorus on the glass walls of the vessel. The formation of the hydrosol must result from the conversion, under the influence of the diffused light, of the dissolved white phosphorus into the red modification, which is almost insoluble in ether, and separates, therefore, at first in the colloidal form. The strong yellow colour of the hydrosol supplies further evidence that the light red colour of the red phosphorus depends on the finely-divided state of the latter. So soon as red phosphorus is deposited on the walls of the vessel, the solution is free from the hydrosol, partly because the colloid is completely precipitated and partly because the deposit protects the remaining white phosphorus from the action of light.

G. Y.

Ionisation by Phosphorus and Phosphorescence. LÉON BLOCH and EUGÈNE BLOCH (*Compt. rend.*, 1908, 147, 842—844).—It has been shown previously (Abstr., 1905, ii, 72) that the air which has passed over phosphorus is the seat of a true ionisation, and the ions produced have a small mobility. It is now found that when an air current of greater velocity is employed, the phosphorescence becomes elongated in the direction of the current, and with a further increase in the velocity, definitely separates itself from the phosphorus with the production of a dark space. The phosphorus generally retains a slight phosphorescence, but this disappears finally when the velocity is sufficiently augmented. If the tube is sufficiently long, the phosphorescence which assumes the form of a phosphorescent column can be removed several metres from the phosphorus by regulating the air-current. A condenser placed in or beyond, but not before, the phosphorescence becomes charged, whilst an iodide-starch paper (test for ozone) becomes blue in the positions in which the condenser is charged. It is thus shown that the phosphorescence, ionisation, and ozone are produced in the same region, and therefore that the three phenomena are produced, not by the oxidation of the phosphorus itself, but of some substance emanating from it. This might be either phosphorus vapour or phosphorous oxide. From the results obtained by Jungfleisch (Abstr., 1905, ii, 244) and by Schenk, Mihr, and Bantien (Abstr., 1906, ii, 326), the latter supposition seems the more probable. This view is also supported by the observation of the authors, that if the dark space is cut suddenly, phosphorescent bubbles are formed, which travel slowly in opposite directions, disappearing either on collision or, more rarely, of their own accord.

If the dark space contains phosphorous oxide, which is spontaneously inflammable, this phenomenon is explained, the movement of the luminous bubbles being the propagation of explosive waves.

The transformation of phosphorus into phosphoric oxide is a vivid combustion, and it is therefore to be expected that the ions produced would be of the same nature as in other cases of combustion. The analogy between the large ions of phosphorus and those of the gases of flames has been pointed out by Bloch and by Harms (Abstr., 1904, ii, 331), who finds an increase in the mobilities as the phosphorus is approached. The latter result is confirmed by the authors, who obtain values up to 0.1 mm. close to the phosphorus. The mobilities can, however, be greatly increased (up to 2–3 mm.) by inserting a cotton plug beyond the phosphorus to retain the fumes formed on the latter. It seems, therefore, that the phosphorus ions are produced by the combustion of the phosphorous oxide, and rapidly rendered heavy by the presence of liquid or solid particles. E. H.

Sublimation of Arsenic. W. P. A. JONKER (*Chem. Weekblad*, 1908, 5, 783–785).—The sublimation point of arsenic determined by the aid of a thermopile is 616°/760 mm. A. J. W.

The Luminosity of the Bunsen Flame. BURRITT S. LACY (*Zeitsch. physikal. Chem.*, 1908, 64, 633–640. Compare Haber and Richardt, Abstr., 1904, ii, 166).—In order to study the cause of the luminosity in the inner zone, the author has introduced methane into a separated Bunsen flame, 5 mm. above the summit of the inner cone, without producing any effect on the luminosity. Special experiments were made to eliminate the cooling effect of the current of methane. The results are in accordance with Haber and Richardt's view, that the production of the light is a luminescence phenomenon. The presence of methane in the middle zone of the separated flame was confirmed.

The constant $k = [\text{H}_2\text{O}][\text{CO}]/[\text{CO}_2][\text{H}_2]$ was found to be the same whether the gases were taken from a zone just above the green cone or from a higher zone at a temperature 300° lower, being about 3.7. A catalytic influence of the platinum tube used was, however, observed.

C. H. D.

Precipitated Silica. HENRY LE CHATELIER (*Compt. rend.*, 1908, 147, 660–662).—The existence of hydrates of silica is questioned on account of the variable amounts of water revealed by analysis. When gelatinous silica, purified from electrolytes by diffusion, was heated in a sealed tube at 320° for six hours, no change in appearance or consistency could be detected. The author draws the conclusion that silica, like chromium trioxide, does not form hydrates, but on account of its absolute insolubility remains in an extremely fine state of division when precipitated. Freshly-precipitated gelatinous silica in distilled water may be used to polish hard substances, such as bronze.

R. J. C.

Products of the Arc and Spark Electric Discharge in Liquid Argon. FRANZ FISCHER and GEORGE ILIOVICI (*Ber.*, 1908, 41, 3802—3810).—If compounds of argon are capable of existence, they are probably endothermic, and might be produced at a high temperature and afterwards be preserved by sudden cooling, as in the formation of ozone in liquid oxygen (compare *Abstr.*, 1907, ii, 163, 340). Cadmium was used for the electrodes, and with the spark discharge, after three hours in liquid argon, 3.3 mg. of an olive-green substance, and with the arc after 140 minutes, 22.7 mg. of a black substance, were obtained. These, on examination, proved to contain cadmium nitride, but mixed with cadmium in the case of the arc product. The spectrum of the gas obtained by heating these substances in a vacuum showed the lines of nitrogen, hydrogen, cadmium, and mercury, with some lines of argon, and the gas obtained by the action of phosphoric acid on the substance gave hydrogen, mercury, and argon lines—nitrogen could not be detected, but the product after the action contained ammonia. The nitrogen must have been derived from small traces of air in the argon used.

The spectroscopic examination was carried out in tubes fitted with aluminium electrodes, previously heated at 150° to expel gases. The tube gradually became "hard," and the nitrogen spectrum disappeared, leaving only the argon and hydrogen lines. This behaviour of the spectrum tube makes the source of the argon uncertain, as it is in very small quantities. The conclusion is drawn provisionally that the argon is absorbed by the nitride.

W. R.

Electrolytic Soda Industry. Theory of the Bell-chamber Process. ANDRÉ BROCHET (*Compt. rend.*, 1908, 147, 674—676).—The salt solution undergoing electrolysis flows through the apparatus from cathode to anode. If the velocity of the liquid is equal or superior to that of the OH⁻ ions, the yield should be theoretical. The velocity of the OH⁻ ion varies directly as the ionic mobility and current density, and inversely as the conductivity. When the layer of liquid containing only OH anions remains stationary, the concentration of the caustic alkali leaving the apparatus varies directly as the conductivity and inversely as the ionic mobility, but is independent of current density and the nature of the cation. It follows that solutions of potassium chloride give a stronger alkali than common brine. Temperature has no influence on the strength of alkali obtained, but a high temperature economises current.

R. J. C.

Transparent Silver and other Metallic Films. THOMAS TURNER (*Proc. Roy. Soc.*, 1908, A, 81, 301—310. Compare Beilby, *Abstr.*, 1904, ii, 647).—The investigations of Faraday on the conditions of formation of transparent metallic films have been extended. Gold leaf does not undergo any change at 500°, but rapidly becomes transparent at 550°, microscopic examination showing that the originally translucent film has become aggregated to opaque masses, leaving clear spaces between them. Soft gold, which has not

been mechanically strained, does not show this sudden change of properties.

Silver foil becomes slightly transparent in air at 240° , and more rapidly at higher temperatures. Heating to 500° in hydrogen or in charcoal powder does not produce transparency, whilst the effect on gold is independent of the surrounding gas. The presence of oxygen is necessary for the change in silver, although the quantity of oxygen absorbed is very minute. The particles of aggregated silver are smaller and less opaque than those of gold.

Copper undergoes a similar change, but much transparent oxide is formed. This is the origin of the coloured surface films obtained on oxidising copper. Aluminium and alloys of copper and zinc do not become transparent when heated.

C. H. D.

The True Atomic Weight of Silver according to Stas's Experiments. LOUIS DUBREUIL (*Compt. rend.*, 1908, 147, 856—859).—It has been shown previously (this vol., ii, 936) that it is generally impossible to deduce the true value of an atomic weight from the measurement of a single analytical ratio, and that the method of least squares enables the most probable value for the atomic weights in question to be determined in each experiment. Comparison of the values so obtained for the same element gives the means of determining the true atomic weight, and the result is the more probable the greater the number of methods employed. The author has applied this method of calculation to the values obtained by Stas for the analytical ratios of AgCl , I , AgI , Ag_2S , KCl , NH_4Cl , NaCl , LiCl , KBr , NaBr , NH_4Br , AgNO_3 , and Ag_2SO_4 , compared with Ag , and for the ratios KCl/AgNO_3 , $\text{NH}_4\text{Cl}/\text{AgNO}_3$, $\text{AgCl}/\text{AgClO}_3$, $\text{AgBr}/\text{AgBrO}_3$, and AgI/AgIO_3 , and, as a general mean of them all, has obtained the value 107.9921 for the atomic weight of silver, the extreme values of the series being 108.0313, obtained from the ratio AgI/Ag , and 107.9449, for the ratio KBr/Ag .

Taking into consideration the fact that all the results employed are taken from the work of one experimenter, the conclusion is drawn that silver has the atomic weight 108.

E. H.

Alloys of Silicon and Silver. G. ARRIVAUT (*Compt. rend.*, 1908, 147, 859—861).—Wöhler (*Ann. Chim. Phys.*, 1858, [iii], 54, 222), Warren (Abstr., 1889, 1125; 1893, ii, 474), and de Chalmot (Abstr., 1896, ii, 362) have obtained substances which they considered to be silver silicides, whereas Percy, Moissan (Abstr., 1896, ii, 173; 1904, ii, 560), and Vigouroux (Abstr., 1907, ii, 543) have repeatedly stated that silver and silicon do not combine. In view of this divergence of opinion, the author has examined mixtures of silver and silicon by Tammann's thermal method. The results are given in a table and in the form of curves.

Examination of the latter shows that the liquidus consists of two branches starting from the melting points of silver (950°) and silicon (1415°) respectively, and intersecting at about 800° in a point corresponding with about 5% of silicon. The solidus consists simply of a horizontal line passing through the eutectic point and extending

from 0% to 90% of silicon; thus silver and silicon do not give any point of combination. Consideration of the periods of eutectic crystallisation suggests the existence of mixed crystals of silicon and silver, but this is not confirmed by analysis. In the fusions containing 40–60% of silicon, a small, white pearl is observed on the bluish-grey button of slightly oxidised silicon. This is formed probably owing to the increase in volume of the silicon on solidification. It contains 4.8% of silicon and 94.85% of silver. E. H.

Silver Sub-halides. A. P. H. TRIVELLI (*Zeitsch. wiss. Photograph. Photophysik. Photochem.*, 1908, 6, 358–372).—A critical comparison of the two chief theories regarding the nature of the silver sub-halides. It is shown that the phenomena, which are usually cited in support of the view that the sub-halide is an adsorption compound of colloidal silver and silver halide, can be explained equally satisfactorily in terms of the theory that the sub-halide is a molecular compound. In certain cases, the molecular theory accounts for the observed facts more satisfactorily than the adsorption theory.

In an appendix, it is pointed out that the reducing action of ammonium persulphate can be explained more simply in terms of the molecular theory than by the adsorption hypothesis. The denser portions of the developed image contain more silver and silver sub-halide and less silver halide than the less dense regions. A sodium thiosulphate solution will, in consequence, be more quickly saturated with silver thiosulphate in the less dense parts of the image. In the denser regions, the greater concentration of unchanged sodium thiosulphate will cause the sub-halide to be decomposed to a greater extent, and the resulting greater concentration of free silver is the cause of the more energetic action of the ammonium persulphate. H. M. D.

Reaction between Silver Sulphide and Silver Sulphate. Production of a Dark-coloured Glass. OTTO SACKUR (*Ber.*, 1908, 41, 3356–3359).—Experiments to determine the dissociation pressure of the system $\text{Ag}_2\text{S} + \text{Ag}_2\text{SO}_4 = 4\text{Ag} + 2\text{SO}_2$ were unsuccessful, as, although a constant pressure was obtainable after heating at a constant temperature (above 300°) for some days, repetition of the experiment gave different values. The pressure at 300° is above 10 atmospheres.

When heated in Thüringian glass, the mixture imparts to the glass surface, below 400° , after some time a reddish-brown colour, which absorbs the actinic rays of the spectrum. Such a glass contains several per cent. of silver. W. R.

Preparation and Composition of the Hydrogen Carbonates of Calcium and Barium. EDWARD H. KREISER and SHERMAN LEAVITT (*J. Amer. Chem. Soc.*, 1908, 30, 1711–1714).—Experiments are described which show that calcium hydrogen carbonate is precipitated when potassium or ammonium hydrogen carbonate is added to a solution of calcium chloride at 0° , and that the salt can be separated and analysed if the temperature is not allowed to rise above 1° or 2° . Even at this temperature, the compound slowly decomposes. The results of analysis point to the formula $\text{CaCO}_3 \cdot 1.75\text{H}_2\text{CO}_3$.

Barium hydrogen carbonate can be obtained similarly by the action of ammonium hydrogen carbonate on barium chloride solution at 0° , but it undergoes gradual decomposition, and is therefore difficult to analyse. E. G.

Composition of the Hydrogen Carbonates of Calcium and Barium. EDWARD H. KEISER and LEROY McMASTER (*J. Amer. Chem. Soc.*, 1908, 30, 1714—1718).—Calcium and barium hydrogen carbonates were prepared in the manner described by Keiser and Leavitt (preceding abstract), except that precipitation was effected in a solution containing gelatin in order to retard decomposition. Analysis was effected by determining the ratio of carbon dioxide to calcium or barium oxide. The results show that the calcium salt has the composition $\text{CaCO}_3 \cdot 1.8\text{H}_2\text{CO}_3$, and the barium salt the composition $\text{BaCO}_3 \cdot 1.5\text{H}_2\text{CO}_3$. E. G.

Preparation of Calcium, Strontium, and Barium Silicides. TH. GOLDSCHMIDT (D.R.-P. 199193).—Calcium silicide may be produced by heating in an ordinary furnace a mixture of silicon and calcium oxide, the reaction being facilitated by the addition of some calcium fluoride and chloride. The interaction takes place as follows: $5\text{CaO} + 5\text{Si} = 2\text{CaSi}_2 + 3\text{CaO} \cdot \text{SiO}_2$. A similar change occurs with the oxides of the other alkaline earths. G. T. M.

Zinc Phosphides. PIERRE JOLIBOIS (*Compt. rend.*, 1908, 147, 801—803).—Zinc and phosphorus were heated together in a crucible until phosphorus vapour ceased to be evolved. The product was freed from excess of zinc either by ignition in a vacuum at 600° or by treatment with mercury or fuming nitric acid. The residual phosphide, Zn_3P_2 , is identical with that described by Vigier (*Bull. Soc. chim.*, 1861, 3, 5), and forms octahedral crystals, $D^{13} 4.55$, which dissolve readily in hydrochloric acid, giving pure phosphine.

The preparation of the phosphide, ZnP_2 , from its elements is also described. This phosphide is non-crystalline, and has $D^{16} 2.97$; at 400° , it undergoes dissociation into phosphorus and Zn_3P_2 . W. O. W.

Sulphides of the Rare Earths. I. Cerium Sulphides and their Limits of Existence. WILHELM BILTZ (*Ber.*, 1908, 41, 3341—3350. Compare Sterba, *Abstr.*, 1904, ii, 662).—*Cerium disulphide*, Ce_2S_4 , is prepared by heating cerium sulphide in a current of hydrogen sulphide at 580 — 600° , the experiment being controlled by weighing from time to time; 2 to 3 grams require twenty hours for complete conversion. It forms a dark yellowish-brown, crystalline powder, moderately stable in air and cold water. An oxygenated product, $\text{Ce}_2\text{S}_{2.5}\text{O}_2$, has also been obtained, and as this is of approximately the same weight as the disulphide, a complete analysis is necessary in characterising the compound. Hydrochloric acid (17.7%) dissolves the disulphide in the cold, an odour of hydrogen persulphide is noticed, and afterwards sulphur is precipitated. This is held to prove that the disulphide is not a true analogue of the dioxide, but a

polysulphide, $\text{Ce}_2\text{S}_3\cdot\text{S}$. The related *lanthanum disulphide*, $\text{La}_2\text{S}_3\cdot\text{S}$, is dark yellow, and also gives hydrogen persulphide under similar conditions.

The red sesquisulphide, Ce_2S_3 (Muthmann and Stützel, Abstr., 1900, ii, 142), is obtained on heating the disulphide or the sulphate in a current of hydrogen sulphide at $750\text{--}800^\circ$.

The conversion of the disulphide into the sesquisulphide has been examined up to 1200° . The temperature of decomposition is about 720° ; the lower sulphide is stable at a red heat, and does not react with hydrogen or nitrogen at that temperature, but between 1400° and 1500° it melts and decomposes. The sesquisulphide prepared at 745° ; when heated in a current of hydrogen sulphide for thirty hours at 615° , did not re-absorb sulphur. This irreversibility is only apparent, as by heating the disulphide in a current of hydrogen at 400° the sesquisulphide was obtained, and this product re-absorbed 4.2% sulphur.

The heat of solution of the disulphide, CeS_2 , in hydrochloric acid is 32,500 cal.; that of $\text{CeS}_{1.5}$, 37,800 cal. (39,500 cal. from product prepared at 400°). From these figures, the equation $4\text{CeS}_2 = 2\text{Ce}_2\text{S}_3 + 2\text{S}(\text{amorp.}) - 18,600 \text{ cal.}$ is calculated.

By the use of Nernst's equation for heterogeneous equilibria, the decomposition temperature is calculated to be 793° , whereas that found was roughly 720° . W. R.

Eutectics. I. The Alloys of Lead and Tin. WALTER ROSENHAIN with P. A. TUCKER (*Phil. Trans.*, 1908, A, 209, 89—122).—Alloys of pure lead and pure tin have been studied by thermal and microscopic methods, the curves of ordinary cooling and of differential cooling being taken. The eutectic point is at 62.33% of tin and 180° . Lead forms solid solutions with from 0 to 16% Sn at 180° . Former observers have found the presence of eutectic in alloys containing much less tin, and this is shown to be due to the slowness with which diffusion takes place in the solid alloys. Heating at 175° for six weeks was found to be necessary to bring about equilibrium. On the other hand, tin does not form solid solutions with small quantities of lead.

Alloys containing from 18 to 63% of tin undergo a change, accompanied by development of heat, on cooling to 149° . In alloys containing from 8 to 18% Sn, the transformation takes place at lower temperatures, the passage of the solid solution from the β - to the α -form involving the rejection of a part of the tin. The alloy with 8% Sn appears to remain in the β -form down to the temperature of liquid air. The lead constituent of the eutectic, however, appears to remain in the metastable β -form. The density of the alloys confirms the view that the lead constituent of the eutectic differs from the stable α -solid solution.

A microscopic study of the eutectic alloy shows that it is composed of grains, within each of which the crystallites have a definite orientation. Each grain is to be regarded as a spherulitic crystal of the β -solution, the tin forming a matrix or filling.

The paper is illustrated with photo-micrographs.

C. H. D.

Electrolytic Corrosion of Brasses in Synthetic Sea-Water. AZARIAH T. LINCOLN and G. C. BARTELS, jun. (*J. Physical Chem.*, 1908, 12, 550—556. Compare Lincoln, Klein, and Howe, *Abstr.*, 1907, ii, 953).—The corrosion of fully annealed copper-zinc alloys was measured, using an artificial sea-water prepared by dissolving salts. The corrosion curves are similar to those obtained in solutions of sodium chloride. With the appearance of the γ -phase, copper ceases to appear in the corrosion product. C. H. D.

Recalculation of the Vapour Pressure of Mercury. T. H. LABY (*Phil. Mag.*, 1908, [vi], 16, 789—796).—The recorded observations of the vapour pressure of mercury have been compared, and the more concordant values have been combined with the object of obtaining a table of most probable values. Two Kirchhoff formulae are given which satisfactorily express the recorded observations. The vapour-pressure values (in terms of mm. of mercury at 0°) are recorded for every 5° between 15° and 365°, and also for 370°, 380°, 390°, 400°, and 450°. It is also shown that the experimental data of Cailletet, Colardeau, and Rivière for temperatures up to 700° can be satisfactorily expressed by means of a Kirchhoff formula. H. M. D.

Constitution of Certain Mercuric Compounds with Complex Cations. II. VINCENZO BORELLI (*Gazzetta*, 1908, 38, ii, 421—474. Compare this vol., i, 515).—According to Abegg and Bodländer's principle of electro-affinity (*Abstr.*, 1899, ii, 542), the tendency of the mercuric halogen salts to form complex cations is weaker the stronger the anion combining with the mercuric ion to form the cation, since increase of the electro-affinity of the halogen radicle should be accompanied by an increase in the tendency of the radicle to dissociate according to the scheme: $\text{HgX}' \rightarrow \text{Hg}'' + \text{X}'$. This is actually found to be the case, the tendency to form these complex ions being greatest with mercuric iodide and cyanide, less marked with the bromide, and so slight with the chloride that the corresponding double salt cannot be isolated. The author has studied the complex salts formed by mercuric iodide, bromide, chloride, and thiocyanate with mercuric perchlorate, the results obtained being parallel with those observed in the case of the similar complex salts yielded by mercuric cyanide. The behaviour of insoluble mercuric compounds of the type HgX'' toward mercuric salts with strong anions is quite analogous to that of the insoluble salts of the type HgX' .

Iodomercury perchlorate, $\text{HgI} \cdot \text{ClO}_4$, forms bundles or mammillary masses of white, opaque prisms, which deliquesce and turn yellow in the air, and are decomposed by water with liberation of mercuric oxide. Cryoscopic measurements in water give values for the molecular weight lower than that calculated for complete dissociation of the compound into its components and allowing for the partial hydrolysis of the mercuric perchlorate according to the equation: $\text{Hg}(\text{ClO}_4)_2 + \text{H}_2\text{O} = \text{Hg}(\text{OH}) \cdot \text{ClO}_4 + \text{HClO}_4$ (compare Heimbucher, *Dis.*, Würzburg, 1904; *Abstr.*, 1904, ii, 465). The depression of the

freezing point of water by mercuric perchlorate is diminished by mercuric iodide, the diminution increasing with the proportion of iodide present in the solution; the complex molecular concentration hence undergoes a corresponding diminution. Similar behaviour is observed when mercuric cyanide is dissolved in an excess of a concentrated solution of mercuric perchlorate. The conductivity of mercuric perchlorate solution is also diminished by the presence in the solution of mercuric iodide, owing to diminution of the ionic concentration and to the lower mobility of the new ions formed. The addition of mercuric iodide or cyanide produces an increase in the molecular concentration of mercuric perchlorate solution when this is not excessively concentrated, and a diminution when an excess of the perchlorate is present. These results are regarded as due to various reactions between the mercuric iodide and the ions, Hg^{++} and ClO_4^- , originally present in the solution. The analogy between these results and those obtained with mercuric cyanide indicates that the mercuric iodide unites with the Hg^{++} ions to give complex cations; this view is supported by transport measurements of solutions of mercuric iodide containing excess of the perchlorate.

Bromomercuric perchlorate, $\text{HgBr}\cdot\text{ClO}_4$, forms bundles or mamillary masses of short, white prisms. When mercuric bromide is dissolved in a solution of mercuric perchlorate, it causes depression of the freezing point and regular elevations of the boiling point, and, consequently, an increase in the concentration. Into these solutions, mercuric bromide enters under the form of the ions HgBr^+ , and also, to a slight extent, of the ions $\text{Hg}_2\text{Br}_2^{++}$. The existence of these complex ions is shown by measurements of the variation of the concentration of mercuric bromide at the electrodes during electrolysis.

Chloromercuric perchlorate could not be isolated. The presence of 1 mol. of mercuric chloride per mol. of the perchlorate in solution produces an increase of 0.028% in the original conductivity, but the addition of a further 1.5 mols. of the chloride causes no further increase. The conductivity curve shows that mercuric chloride exhibits a tendency to form the ions HgCl^+ about 500 times less than the tendency of the cyanide to form the ions HgCN^+ .

The author has not succeeded in isolating chloromercuric nitrate, $\text{HgCl}\cdot\text{NO}_3$ (compare Morse, Abstr., 1903, ii, 12), but conductivity measurements demonstrate the undoubted presence of the ions HgCl^+ and NO_3^- in solutions containing mercuric chloride and nitrate.

Thiocyanomercuric perchlorate, $\text{Hg}(\text{CNS})\cdot\text{ClO}_4$, was obtained as a white, crystalline crust. The addition of mercuric thiocyanate to a solution of mercuric perchlorate produces a diminution in the molecular concentration.

The author has been unable to prepare the compound, $2\text{Hg}(\text{ClO}_4)_2\cdot\text{HgO}\cdot 12\text{H}_2\text{O}$, described by Chikashigé (Trans., 1905, 87, 822), the products obtained being basic compounds containing approximately $2\text{Hg}(\text{ClO}_4)_2$ per 3HgO . When mercuric oxide is dissolved in a solution of mercuric perchlorate, it produces diminution of the molecular concentration; the diminution produced increases with the concentration

of the perchlorate solution, as, therefore, does the complexity of the compounds formed.

T. H. P.

Precipitation of Gelatinous Mixtures of Alumina and Silica, and their Relation to Allophane, Halloysite, and Montmorillonite. H. STREMMER (*Centr. Min.*, 1908, 622—632, 661—669).—The gelatinous precipitates obtained from solutions of sodium silicate and aluminium acetate are of very variable composition, depending on the strength of the solutions and on their acidity. In an acid solution there is at first only a turbidity, and the gelatinous precipitate afterwards deposited is richer in silica than that from a neutral solution.

Carbonic acid and acetic acid dissolve the alumina more readily than the silica from the precipitates. The conclusion is drawn that these precipitates do not represent any definite hydrated aluminium silicate, but that they are simply mixtures of gelatinous aluminium hydroxide and hydrated silica. The same relations also exist in the various amorphous minerals of the clay group. The published analyses of these are tabulated, and they show wide variations in composition: for minerals of the allophane group, the ratio $\text{Al}_2\text{O}_3:\text{SiO}_2$ varies from 1:0.31 to 1:1.91; for the halloysite group, 1:1.68 to 1:3.89; and for the montmorillonite group, 1:2.51 to 1:5.32. The conclusion is therefore drawn that these are not definite mineral species, but mixtures in variable proportions of colloidal aluminium hydroxide and hydrated silica.

L. J. S.

Ferromagnetic Nitrogen Compounds of Manganese. EDGAR WEDEKIND and THEO. VEIT (*Ber.*, 1908, 41, 3769—3773. Compare Abstr., 1907, ii, 353; Shukoff, this vol., ii, 484).—With the object of ascertaining whether elements which are gases at the ordinary temperature form ferromagnetic derivatives with manganese, the nitrides of manganese have been investigated. Trimanganese nitride, Mn_3N_2 (compare Prelinger, Abstr., 1894, ii, 16), has only feeble magnetic properties, whilst pentamanganese nitride, Mn_5N_2 , is slightly more magnetic. The nitride, Mn_7N_2 , obtained by the action of ammonia on manganese heated with an oxy-hydrogen blowpipe, has stronger magnetic properties than either of the above nitrides, and is almost as magnetic as manganese boride; it is readily attacked by acids and alkalis.

Chromium nitride, CrN , has only feeble magnetic properties.

W. H. G.

The System Iron-Phosphorus. E. GERCKE (*Metallurgie*, 1908, 5, 604—609).—Alloys rich in phosphorus were prepared by adding red phosphorus to molten iron in a magnesite crucible. The product containing 17.3% P was re-melted with different proportions of iron. The eutectic temperature is 980° ; mixtures low in phosphorus show considerable undercooling. Iron retains up to 1.70% of phosphorus in solid solution, beyond this the eutectic, containing 10.2% P, is present as a constituent. The solid solution undergoes a change of structure, which has not been fully investigated, at 600° .

C. H. D.

The Ternary System Iron-Phosphorus-Carbon. PAUL GOERENS and W. DOBBELSTEIN (*Metallurgie*, 1908, 5, 561—566. Compare Wüst, this vol., ii, 287).—The ternary eutectic point of the system is found to lie at 1.96% C, 6.89% P, and 91.15% Fe, and at 953°. The position of the three binary eutectic curves in the system iron-iron carbide-iron phosphide has also been determined. Both thermal and microscopic methods have been employed.

The microscopic sections are best prepared by etching and heat-tinting, subsequently re-polishing lightly. The oxide film is much more readily removed from cementite than from the phosphide, thus allowing the two to be distinguished. C. H. D.

The Iron-Carbon Equilibrium. GEORGE B. UPTON (*J. Physical Chem.*, 1908, 12, 507—549).—The results of other observers are examined and recalculated, the silicon and other elements present in the samples examined being calculated as "equivalent carbon." The author concludes that cementite must find a place in the diagram of stable equilibrium, and he assumes the formation of three carbides in all, Fe_3C , Fe_2C , and Fe_7C_3 , of which the first is stable only above 800° and the second above 600°. The brittleness of steel at 500—550° is considered to be due to the change from Fe_3C to Fe_2C . An equilibrium diagram has been constructed on these assumptions. C. H. D.

Hydrolysis of Ferric Chloride; Influence of Neutral Salts. G. MALFITANO and LEOPOLD MICHEL (*Compt. rend.*, 1908, 147, 803—806. Compare Abstr., 1907, ii, 692; this vol., ii, 111, 288).—The phenomena of the colloidal state are more pronounced and appear more rapidly in solutions of ferric chloride to which potassium chloride has been added than in solutions of ferric chloride alone. The authors have measured the increase in electrical conductivity of solutions of ferric chloride, alone and with varying amounts of potassium chloride, produced by raising the temperature from 18° to 100° for fifteen minutes, and after allowing the solutions to remain for sixteen and twenty-four hours respectively. The results lead to the conclusion that the rate of hydrolysis of the ferric chloride depends on the proportion of potassium chloride present, and that the size of the micro-cells of ferric hydroxide is increased by the addition of this salt. The number of ferric ions being diminished, there are fewer centres of attraction for the ferric hydroxide molecules, and consequently the individual cells are more voluminous.

The chlorides of sodium, ammonium, barium, and magnesium, and also potassium nitrate, behave in a similar manner. Mercuric chloride, on the other hand, being a non-electrolyte, has no appreciable influence on the course of the hydrolysis. Salts containing multivalent negative ions, such as potassium sulphate, behave like the multivalent acids previously studied in hindering hydrolysis. W. O. W.

Molybdates of Nickel and Cobalt. M. EMMANUEL POZZI-ESCOR (*Bull. Soc. chim.*, 1908, [iv], 3, 1012).—The author has been informed by Marckwald that the attribution to the latter by Grossmann and

Schück (this vol., ii, 230) of certain work on nickel and cobalt molybdates is inaccurate. T. A. H.

Antimony Trichloride as Ionising Solvent. Z. KLEMENSIEWICZ (*Bull. Acad. Sci. Cracow*, 1908, 485—494).—The measurements were made by the electrical conductivity method with platinised electrodes in the usual way. The carefully-purified solvent has D_{25}^0 2.681 and D_{27}^0 2.647; the smallest conductivity observed amounted to 8.5×10^{-7} reciprocal ohms.

As solutes, the chlorides of potassium, rubidium, ammonium, and thallium were used, and measurements were made up to a dilution of 1024 litres and at different temperatures. The conductivities in dilute solutions are greater, in concentrated solutions (above $N/10$) less, than for the corresponding aqueous solutions. It is probable that the degree of ionisation is less for solutions in antimony chloride than in water, but that the ionic velocity is greater in the former case. The conductivity of the solutions increases regularly with the temperature from 70° to 150 — 200° .

The viscosity of antimony trichloride has been measured from 80° to 200° . The curve obtained by plotting the fluidity (reciprocal of the viscosity) against the temperature appears to show a change of direction at 120° , but a high degree of accuracy is not claimed for the results. G. S.

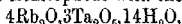
Melting-point Curves for the Systems Bismuth + Chlorine and Bismuth + Bromine. B. G. EGGINK (*Zeitsch. physikal. Chem.*, 1908, 64, 449—505).—After reviewing the various compounds of bismuth with chlorine and bromine which have been described, the author proceeds to a theoretical discussion of the possible liquid-solid equilibria in a system of two components in which two liquid phases may occur. In this discussion, the author uses the graphical method introduced by van Ryn van Alkemade (*Abstr.*, 1893, ii, 363).

The experimental study of the systems bismuth + chlorine and bismuth + bromine has shown that compounds of the formulæ BiCl and BiBr exist, which, on melting at 320° and 287° respectively, both form two liquid phases. Further, an endothermic compound BiCl_4 exists, which is only slightly dissociated in the fused condition. So far as the evidence of the freezing-point curve goes, there are no compounds of the formulæ BiCl_2 , BiBr_2 , and Bi_3Cl_8 ; the author's results are therefore not in harmony with those of Herz and Guthmann (this vol., ii, 199). J. C. P.

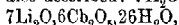
Columbium. CLARENCE W. BALKE and EDGAR F. SMITH (*J. Amer. Chem. Soc.*, 1908, 30, 1637—1668).—A continuation of the investigation of the compounds of columbium and tantalum (*Abstr.*, 1905, ii, 828, 829). The conclusion is confirmed that the coloration produced on the addition of hydrogen peroxide to a solution of columbium in hydrofluoric acid is not due to the presence of titanium, but is probably due to columbium itself. A large quantity of potassium columbium fluoride, K_2CbF_7 , was prepared and carefully purified, and was after-

wards converted into the oxide, which had D 4.8. This oxide still gave the reaction with hydrogen peroxide, and, in order to study further the question as to whether the reaction might not be due to the presence of some other element, the oxide was converted into the chloride, and the latter fractionally distilled. The whole of the product distilled at about 241° , and no differences could be detected between the fractions obtained. On a spectroscopic examination of the oxide prepared from the chloride, neither tantalum nor titanium lines were observed. Vapour density determinations of the chloride by Dumas' method gave a mean value of 9.45, as compared with the calculated value 9.35 [Cb = 93.5]. Specimens of the oxide prepared from the chloride had D varying from 4.48 to 5.02. The chloride had D 2.73–2.77. A careful determination of the atomic weight of columbium based on the ratio between the chloride and oxide gave as mean of eight determinations the value 93.50 [O = 16; Cl = 34.45].

A résumé is given of the various columbates described in the literature. Sodium columbate, $\text{Na}_2\text{O} \cdot \text{Cb}_2\text{O}_5 \cdot 7\text{H}_2\text{O}$, crystallises in triclinic prisms [$a : b : c = 0.9559 : 1 : 0.8394$; $\alpha = 71^{\circ}20'$; $\beta = 105^{\circ}30'$; $\gamma = 54^{\circ}7'$]. Magnesium columbate, $\text{MgO} \cdot \text{Cb}_2\text{O}_5 \cdot 7\text{H}_2\text{O}$, the silver salt, $\text{Ag}_2\text{O} \cdot \text{Cb}_2\text{O}_5 \cdot 2\text{H}_2\text{O}$, the copper salt, $\text{CuO} \cdot \text{Cb}_2\text{O}_5 \cdot 3\frac{1}{2}\text{H}_2\text{O}$, the aluminium salt, $\text{Al}_2\text{O}_3 \cdot 3\text{Cb}_2\text{O}_5 \cdot 12\text{H}_2\text{O}$, and the cadmium salt, $\text{CdO} \cdot \text{Cb}_2\text{O}_5 \cdot 3\frac{1}{2}\text{H}_2\text{O}$, are described. The potassium salt, $4\text{K}_2\text{O} \cdot 3\text{Cb}_2\text{O}_5 \cdot 16\text{H}_2\text{O}$, forms monoclinic crystals [$a : b : c = 0.7120 : 1 : 0.5547$; $\beta = 84^{\circ}19'$]. The rubidium salt, $4\text{Rb}_2\text{O} \cdot 3\text{Cb}_2\text{O}_5 \cdot 14\text{H}_2\text{O}$, is isomorphous with the salts



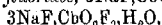
$4\text{Cs}_2\text{O} \cdot 3\text{Cb}_2\text{O}_5 \cdot 14\text{H}_2\text{O}$, and $4\text{Cs}_2\text{O} \cdot 3\text{Ta}_2\text{O}_5 \cdot 14\text{H}_2\text{O}$, and crystallises in monoclinic prisms [$a : b : c = 0.8815 : 1 : 1.4091$; $\beta = 84^{\circ}7'$]. The following columbates are also described: $7\text{K}_2\text{O} \cdot 6\text{Cb}_2\text{O}_5 \cdot 27\text{H}_2\text{O}$,



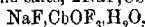
$7\text{Cs}_2\text{O} \cdot 6\text{Cb}_2\text{O}_5 \cdot 30\text{H}_2\text{O}$, and $3\text{Rb}_2\text{O} \cdot 4\text{Cb}_2\text{O}_5 \cdot 9\frac{1}{2}\text{H}_2\text{O}$.

The following percolumbates have been obtained: Na_2CbO_5 , K_3CbO_8 , Rb_3CbO_8 , Cs_3CbO_8 , $\text{MgNaCbO}_8 \cdot 8\text{H}_2\text{O}$, $\text{MgKCbO}_8 \cdot \text{H}_2\text{O}$, $\text{MgRb}_2\text{CbO}_8 \cdot 7\frac{1}{2}\text{H}_2\text{O}$, $\text{MgCsCbO}_8 \cdot 8\text{H}_2\text{O}$, $\text{CaNaCbO}_8 \cdot 4\text{H}_2\text{O}$, and $\text{CaKCbO}_8 \cdot 4\text{H}_2\text{O}$.

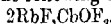
The sodium columbium fluorides, $3\text{NaF} \cdot \text{CbOF}_3$ and



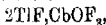
have been prepared, but the salts, $2\text{NaF} \cdot \text{CbOF}_3 \cdot 2\text{H}_2\text{O}$ and



described by Marignac could not be obtained. The existence of the potassium salts, $2\text{KF} \cdot \text{CbOF}_3 \cdot \text{H}_2\text{O}$, $3\text{KF} \cdot \text{CbOF}_3$, $3\text{KF} \cdot \text{HF} \cdot \text{CbOF}_3$, $5\text{KF} \cdot 3\text{CbOF}_3 \cdot \text{H}_2\text{O}$, $4\text{KF} \cdot 3\text{CbOF}_3 \cdot 2\text{H}_2\text{O}$, and $2\text{KF} \cdot \text{CbF}_5$, described by Marignac, was confirmed. The salt $3\text{KF} \cdot \text{HF} \cdot \text{CbOF}_3$ forms monoclinic needles [$a : b : c = 0.6304 : 1 : 0.4888$; $\beta = 86^{\circ}41'$]. The double fluoride, $2\text{RbF} \cdot \text{CbF}_3$, described by Pennington (Abstr., 1896, ii, 305) could not be obtained, but the following salts were prepared:



$\text{RbF} \cdot \text{CbF}_5$, and $2\text{RbF} \cdot \text{CbO}_2\text{F}_3 \cdot \text{H}_2\text{O}$. The rubidium tantalum salt, $2\text{RbF} \cdot \text{TaO}_2\text{F}_3 \cdot \text{H}_2\text{O}$, forms thin, white leaflets. The caesium salts, $2\text{CsF} \cdot \text{CbOF}_3$ and $\text{CsF} \cdot \text{CbF}_5$, are described. The thallium salt,



forms orthorhombic crystals [$a : b : c = 0.4261 : 1 : 1.0129$].

A study has been made of the double tantalates. The salt
 $4\text{Rb}_2\text{O} \cdot 3\text{Ta}_2\text{O}_5 \cdot 14\text{H}_2\text{O}$
 forms monoclinic crystals [$a:b:c=0.8822:1:1.0510$; $\beta=84^\circ 2'$].
 The salts $4\text{Cs}_2\text{O} \cdot 3\text{Ta}_2\text{O}_5 \cdot 14\text{H}_2\text{O}$ and $7\text{Cs}_2\text{O} \cdot 6\text{Ta}_2\text{O}_5 \cdot 38\text{H}_2\text{O}$ are also
 described.

The following *pertantalates* have been prepared: Rb_3TaO_8 , Cs_3TaO_8 ,
 $\text{MgNaTaO}_8 \cdot 8\text{H}_2\text{O}$, $\text{MgKTaO}_8 \cdot 7\text{H}_2\text{O}$, $\text{MgRbTaO}_8 \cdot 9\text{H}_2\text{O}$, and
 $\text{CsNaTaO}_8 \cdot 4\frac{1}{2}\text{H}_2\text{O}$.

E. G.

Spectrum and the Bromides of Columbium. WILLIAM M. BARR (*J. Amer. Chem. Soc.*, 1908, 30, 1668—1672).—It has been shown by Hall and Smith (Abstr., 1905, ii, 829) that the hydrogen peroxide test for titanium in a solution of columbium in hydrofluoric acid is not trustworthy. For this reason, spectroscopic examination has been resorted to. Hildebrand (following abstract) has submitted carefully-purified columbium oxide prepared by Balke and Smith (preceding abstract) to a spectroscopic examination, and has found that, whilst nearly all the titanium lines were absent from the spectrum, certain lines were present which are common to both metals. A study has therefore been made of the spectra of specimens of columbium oxide prepared from minerals of different character and from different localities, and purified by Balke and Smith's methods. The results indicate that these methods of purification are efficient, that the spectra of columbium from all sources are identical, and that the lines common to both the columbium and titanium spectra are not due to the presence of titanium in the columbium, and probably not to any other element, but are merely coincident lines.

The "Niobunterbromid" described by Rose (*Ann. Phys. Chem.*, 1858, 104, 441) has been studied, and found to be columbium oxybromide, CbOBr_3 . The dark red compound formed simultaneously is the pentabromide, CbBr_5 , as stated by Rose. Both these salts are described.

A columbium iodide has been prepared from the bromide, and is being investigated.

E. G.

Arc Spectrum of Columbium. JOEL H. HILDEBRAND (*J. Amer. Chem. Soc.*, 1908, 30, 1672—1684. Compare Balke and Smith, and Barr, preceding abstracts).—The arc spectrum of columbium has been measured between $\lambda 2600$ and $\lambda 6000$, using specimens of the oxide obtained from euxenite, tantalite, columbite, and aeschynite. The results are tabulated.

It has been found that Balke and Smith's method for preparing columbium oxide, free from titanium, is satisfactory, and that the existence of any element common to columbium and titanium is very improbable. The identity of the spectra of columbium from different sources affords strong evidence of its elementary character.

E. G.

Metallographic and Metallurgical Notes. [Thermal Diagrams. Microscopy of Alloys.] K. FRIEDRICH (*Metallurgie*, 1908, 5, 593—604).—I. It was found impossible to obtain thermal

diagrams of the systems platinum-sulphur, gold-selenium, gold-sulphur, gold-arsenic, or zinc-sulphur on account of the volatility of one component. The existence of the sulphide, Zn_2S , is improbable.

II. The use of ultra-violet light in the microscopic study of alloys is not found to have any advantages.

C. H. D.

Atomic Weight of Palladium. GEORGE I. KEMMEER (*J. Amer. Chem. Soc.*, 1908, 30, 1701—1705).—The results of previous work on the atomic weight of palladium show considerable variation, and, for this reason, the present investigation was undertaken. The experiments were carried out with (A) palladodiammonium chloride purified by Keller and Smith's method (*Abstr.*, 1893, ii, 73); (B) the same salt purified by means of ammonium cyanide instead of the mercuric cyanide used by Keller and Smith, and ammonium salts instead of sodium or potassium salts, and (C) palladodiammonium cyanide, $\text{Pd}(\text{NH}_3\text{CN})_2$. In each case a porcelain boat containing the salt was enclosed in a tube heated by an electric heater, and was reduced to the metal by means of carefully-purified electrolytic hydrogen. The following results were obtained: salt A, mean of five experiments, 106.399; salt B, mean of four experiments, 106.442; salt C, mean of six experiments, 106.458. The mean value of all the determinations gave an atomic weight, 106.434 [$\text{Cl}=35.473$; $\text{H}=1.008$; $\text{N}=14.01$].

E. G.

Mineralogical Chemistry.

Natural Zirconium Oxide Free from Iron. EDGAR WEDEKIND (*Zeitsch. angew. Chem.*, 1908, 21, 2270—2271).—Of the three forms in which Brazilian zirconium ore occurs, that containing the greatest amount of zirconium is the black, glassy, lump variety; analysis gave:

ZrO_2	TiO_2	Fe_2O_3	SiO_2	ZrSiO_4	Total.
94.12	0.98	3.22	0.43	1.98	100.73

When examined under the microscope, this mineral was found to be composed of black particles embedded in a brownish-yellow, powdery substance. When separated mechanically, the black portion was found to be quite free from iron; analysis gave:

ZrO_2	SiO_2	TiO_2	Insol.	Total.
97.97	1.72	1.20	0.10	100.99

From this it is evident that the colour previously ascribed to the presence of iron must be caused by the titanium, as, indeed, must be the colour of many other stones and minerals. Crystalline, natural zirconium oxide is feebly radioactive; it has D 5.41, a value somewhat higher than that, D 5.1, of the artificial, crystalline substance.

J. V. E.

Kröhnkite, Natrochalcite (a New Mineral), and Other Sulphates from Chile. CHARLES PALACHE and CHARLES H. WARREN (*Amer. J. Sci.*, 1908, [iv], 26, 342—348 *).—The specimens described are from copper veins in the mining district of Chuquicamata, in the province of Antofagasta, and include kröhnkite, natrochalcite, blödite, brochantite, atacamite, chalcantinite, copiapite, botryogen, sideronatrite, halite, and gypsum.

Kröhnkite.—The large, well-developed crystals are monoclinic, with $a : b : c = 0.5229 : 1 : 0.4357$; $\beta = 56^{\circ}17\frac{1}{2}'$; they are twinned on (001), and have a perfect cleavage parallel to (010); hardness $2\frac{1}{2}$. Three types of specimens are distinguished: (i) clusters of octahedroid crystals of a dull greenish-blue colour; (ii) single prismatic crystals and fibrous or acicular aggregates of a pale blue colour; (iii) solid crusts of large, prismatic crystals of a deep vitriol-blue colour. Analysis I gives the usual formula: $\text{CuSO}_4 \cdot \text{Na}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$. The water is mainly given off below 150° , but small amounts continue to come off up to 350° . The mineral is fusible without decomposition to a bright green enamel.

Natrochalcite.—This new species occurs as bright emerald-green, monoclinic crystals with an acute pyramidal habit [$a : b : c = 1.423 : 1 : 1.214$; $\beta = 61^{\circ}17\frac{1}{2}'$]. Cleavage (001) perfect; hardness $4\frac{1}{2}$. Complete optical determinations are given for this, as also for kröhnkite. Analysis II agrees with $\text{Na}_2\text{SO}_4 \cdot \text{Cu}_4(\text{OH})_2(\text{SO}_4)_2 \cdot 2\text{H}_2\text{O}$. The water is given off gradually above 150° , and between 350° and a low red-heat, sulphuric anhydride is expelled. The mineral readily fuses to a black bead.

Blödite.—Analysis III, of massive, granular material, agrees with the usual formula: $\text{MgSO}_4 \cdot \text{Na}_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$.

	CuO.	MgO.	Na ₂ O.	SO ₃ .	H ₂ O.	Insol.	Cl.*	Total.	Sp. gr.
I.	23.25	—	18.89	47.60	10.72	—	trace	100.46	2.061
II.	41.95	—	8.44	42.10	7.70	0.70	0.05	100.94	2.33
III.	—	12.00	18.20	47.49	21.60	0.50	—	99.70	—

* Cl from atacamite.

L. J. S.

Analyses of Gabbroitic Rocks from Neurode, Silesia. FELIX TANNHÄUSER (*Sitzungsber. k. Akad. Wiss. Berlin*, 1908, 1069—1075).—Nine analyses are given of gabbro, anorthosite, pyroxenite, olivine-gabbro, troctolite, serpentine, diabase, gabbro-aplite, and spessartite.

L. J. S.

Physiological Chemistry.

Heats of Solution of Gases in Blood. MARIO CAMIS (*Mem. Real. Accad. Sci. Torino*, 1908, [ii], 58, 141—169).—The author has measured calorimetrically the heat changes occurring when oxygen and carbon dioxide pass into, or out of, solution in blood. The absorption of 1 gram-mol. of carbon dioxide by the blood is accompanied by the evolution of 5.40 Cal., the corresponding value for oxygen being

* and *Zeitsch. Kryst. Min.*, 1908, 45, 529—538.

10-22 Cal. Taking mean numbers for the daily respiratory exchange, it is calculated that, for a man weighing 70 kilos., this exchange leads to the production of about 127 Cal. per day. The blood pigment and the oxygen absorbed probably react endothermically. The paper concludes with a bibliography. T. H. P.

Estimation of the Respiratory Capacity of Small Quantities of Blood. HEINRICH DRESER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 138—149).—Certain modifications of Haldane's carbon monoxide method, and of Barcroft's gas analysis methods, are proposed. In investigating the effect of certain antipyretics on the respiratory capacity, it was found that many of these produce methæmoglobin formation, and the oxygen so combined is not displaced by carbon monoxide. A comparison of *m*-tolylhydrazine, β -acetylphenylhydrazine (pyrodine), and the semicarbazide of *m*-tolylhydrazine shows that the semicarbazide substitution is much less harmful than the acetyl substitution, as in acetanilide and phenacetin. W. D. H.

Glycine in Normal Blood. ADOLF BINGEL (*Zeitsch. physiol. Chem.*, 1908, 57, 382—388).—By the use of the Fischer-Bergell naphthalenesulphonyl chloride method, it is possible to demonstrate the existence of glycine in normal ox-blood. From 10 litres, 0.2 gram of the glycine compound was obtained. A higher amino-acid, or a peptide-like substance, is also present. W. D. H.

Reaction of the Blood after Intravenous Injection of Acid and Alkali. N. VAN WESTENRYK (*Arch. exp. Path. Pharm., Suppl.*, 1908, 517—527).—That the titration method shows the blood to be alkaline is due to the fact that the acid used may displace a weaker acid in neutral salts. Many indicators are not indifferent chemical substances, but may be themselves acid or basic. By the use of neutral-red and other indicators, the reaction of the blood is shown to be neutral, and remains neutral under various pathological conditions. It alters in experimental acid or alkali poisoning. Alkali poisoning produces stimulation, increasing the action of the heart and kidneys; acid produces the opposite effects. In acid poisoning, the organism remains neutral, as the acid is fixed and neutralised in the muscular tissues. Previous observations that the alkalinity of the blood decreases on destruction of the red corpuscles, are confirmed by the new methods. W. D. H.

Spectrophotographic Investigations on the Action of Hydrocyanic Acid on Blood. LOUIS LEWIN (*Arch. exp. Path. Pharm., Suppl.*, 1908, 337—348).—Details are given of the spectroscopic appearances of blood and hæmoglobin treated with hydrocyanic acid. Cyanomethæmoglobin and cyanohæmatin do not appear to exist; the spectroscopic appearances of their so-called compounds are identical with those of cyanohæmoglobin. Blood so treated catalyses hydrogen peroxide quite typically. The cause of the toxic action of hydrocyanic acid is discussed, and the conclusion is drawn that it is not a blood poison in the biological sense. W. D. H.

Action of Oxidising Salts. ARTHUR R. CUSHNY (*Arch. exp. Path. Pharm., Suppl.*, 1908, 126—137).—The relative activity of oxidising salts on the blood and tissues runs fairly parallel to their oxidising power on simpler chemical compounds. But in many cases other factors step in. The blood corpuscles and pigment react more readily to weak oxidising agents than do muscle or epithelium. In certain cases, also, substances which oxidise blood corpuscles and pigment readily are feeble oxidising agents for simpler chemical substances; the reverse is also seen.

W. D. H.

Hæmolytic Action of Mercury Salts. J. DUNIN-BORKOWSKI (*Bull. Acad. Sci. Cracow*, 1908, 494—505).—The hæmolytic action of mercury salts on the serum-free blood corpuscles (erythrocytes) of different animals has been investigated. With mercuric chloride, the resistance to hæmolysis increases in the order: guinea-pig, sheep, cow, pig, calf, dog. A higher concentration of mercuric chloride than that required to produce complete hæmolysis, agglutinates the erythrocytes.

Mercuric iodide has considerable hæmolytic activity, and mercuric fluoride, although not ionised, is almost as active as the chloride. Potassium cyanide is less active than mercuric cyanide. It follows that hæmolysis does not depend on the concentration of Hg^{++} ions alone, although the ionic concentration probably has some effect, as sodium chloride lessens the activity of mercuric chloride.

The rate of hæmolysis with different concentrations of mercuric chloride has also been measured. With small concentrations, the constants calculated for a unimolecular reaction diminish, and with fairly high concentrations they increase considerably during the reaction, being approximately constant for intermediate concentrations.

The temperature-coefficient for 10° between 18° and 25° is 4.37 for erythrocytes from the guinea-pig, and 2.6 between 18° and 32° for those from the dog. The coefficient is much greater at higher temperatures, owing to the fact that hæmolysis is produced to some extent by heat alone, and, further, the erythrocytes which have been heated alone for some time are much more readily acted on by mercuric chloride than those which have not been heated.

G. S.

Peptolytic Ferments in the Stomach Contents. EMIL BERGHAARDEN and FLORENTIN MEDIGRECEANU (*Zeitsch. physiol. Chem.*, 1908, 57, 317—324).—The observations were made on a dog with gastric fistula, and confirm those made by Boldyreff, that, after a meal rich in fat, the contents of the duodenum regurgitate into the stomach. Under these conditions, the stomach contents contain a peptolytic ferment, and when neutralised, resolve glycyl-L-tyrosine into its constituent amino-acids. Under ordinary conditions, however, the ferment is rendered rapidly inactive by the acid of the gastric juice.

W. D. H.

Influence of the Products of Reaction on the Hydrolysis of Fats by Pancreatic Juice. Mlle. L. KALABOUKOFF and Émile TERROINE (*Compt. rend.*, 1908, 147, 712—715).—Both fatty acids and their sodium salts diminish the rate of hydrolysis; glycerol, on the other hand, has a considerable accelerating action. This acceleration does not take place when a soluble ester, such as monobutyryl, or natural emulsions, such as egg-yolk or cream, or solid fats, such as lard, are submitted to the action of the juice. The action of the glycerol is to be ascribed to the fact that a better distribution of the lipase between the aqueous and oily phases takes place in its presence; other syrupy substances can produce a similar acceleration.
S. B. S.

Digestion in Animals. XXVII. Relationship of Concentration to Absorption in the Intestine. E. S. LONDON and W. W. POLOWZOWA (*Zeitsch. physiol. Chem.*, 1908, 57, 529—546. Compare this vol., ii, 960).—Details are given of the relative amount of absorption in the intestine of the water and sugar contained in solutions of dextrose of different concentrations.
W. D. H.

Absorption of Fat from Intestinal Loops in Dogs. O. H. PLANT (*Amer. J. Physiol.*, 1908, 23, 65—80).—Bile salts increase the absorption of fats from a mixture which contains free fatty acid or soap; they only slightly increase the absorption of neutral oil. Solutions of soap, or biliary solutions of fatty acids, are absorbed more rapidly than emulsified fats. If both bile and pancreatic juice are excluded from the intestinal loop, neutral oil is nevertheless absorbed, and it becomes markedly acid in reaction. Taken as a whole, the experiments favour the view that fats are absorbed in solution rather than as an emulsion.
W. D. H.

Absorption of Iodised Proteins. OTTO VON FÜRTH and M. FRIEDMANN (*Arch. exp. Path. Pharm., Suppl.*, 1908, 214—223).—An iodised protein (iodalbacid) is before absorption in the cat's intestine broken down in large measure so completely that the iodine in the intestinal wall and blood is not in combination as proteose or peptone, but only as alkali iodides.
W. D. H.

Action of Intestinal Astringents on Metabolism. KARL SPIRO (*Arch. exp. Path. Pharm., Suppl.*, 1908, 504—512).—Opium, bismuth subnitrate, and tannigen produce but little change in nitrogenous metabolism in dogs. With opium the C:N ratio in the urine goes up, this is due to a fall in the nitrogen; the nitrogen in the faeces is but little altered. Tannigen, on the other hand, causes a loss of urinary, and an increase of faecal nitrogen, the total excretion of nitrogen being about the normal; a sinking of the C:N ratio in the urine is mainly due to a relative decrease in the excretion of carbon.
W. D. H.

Creatinine Metabolism. G. LEFMANN (*Zeitsch. physiol. Chem.*, 1908, 57, 476—514).—The excretion of creatinine and creatine is

pretty constant in well nourished animals. If either substance is added to the food, it is excreted unchanged. If creatine is given by the mouth or parenterally, it is never changed into creatinine; in inanition it is almost completely excreted as such. Disease of the liver or increased protein catabolism produce first an increase, then a decrease, in creatinine excretion, and when it is lessened, the amount of creatine excreted increases. The liver is the probable seat of creatinine formation. If nephritis is induced by chromates, nearly all the creatinine is changed into creatine, probably by the alteration in the reaction of the urine.

W. D. H.

The Changes in Gaseous Metabolism after Exclusion of the Hepatic Circulation. VITTORIO SCAFFIDI (*Biochem. Zeitsch.*, 1908, 14, 156—179).—Experiments were carried out on ducks, the liver circulation being excluded by ligaturing the portal vein. As birds were employed for the experiments, it was not necessary to make an Eck fistula. The absorption of oxygen and excretion of carbon dioxide were determined both before and after the operation. It was found that the operation caused an increase in the oxygen absorption and in the carbon dioxide excretion, and also an increased respiratory quotient. These results are probably due to the inhibition of glycogen storage, owing to the liver being thrown out of circulation. There is consequently an increased destruction of the carbohydrates, which are thrown into the circulation. The increased respiratory quotient, which is particularly noticeable at some interval after the operation, is probably due to the conversion of the carbohydrate into fat.

S. B. S.

Starvation Metabolism. MIECZYSLAW HALPERN (*Biochem. Zeitsch.*, 1908, 14, 134—142).—The urine of a patient unable to ingest either food or water, owing to cancer in the oesophagus, was analysed. The total daily excretion of nitrogen was 2.058 grams, or, excluding the protein nitrogen excreted, 2.0097 grams. This is less than found in most other starvation cases, owing probably to the fact that the organism had gradually accommodated itself to a low diet. Other anomalies were also observed, for the ammonia nitrogen was only 4.7% of the total. The excretion of the acetone substances was also small. The purine substances were also small in amount, the daily excretion of purine nitrogen being only 0.05897 gram. The sodium chloride excretion was 0.05265 gram daily, whilst the ratio $N : P_2O_5$ was 6.3 : 1, which is normal and higher than in other starvation cases. The author discusses the possible reasons for the anomalies.

S. B. S.

The Nutritive Value of Protein Cleavage Products. VIII. HILF ABDERHALDEN (*Zeitsch. physiol. Chem.*, 1908, 57, 348—362. Compare this vol. ii, 961).—Dogs react differently by feeding on protein cleavage products, some being attacked with vomiting. But those which do not react in this way, equilibrium and health are well maintained. Complete details of the experiments are tabulated. The same result was obtained also with the products of acid hydrolysis.

of flesh and casein. If, however, the greatest part of the tryptophan and tyrosine was removed from the mixture beforehand, the results were not so good.

W. D. H.

Phosphorus in Certain Foods WOLFGANG HEUBNER and M. REEB (*Arch. exp. Path. Pharm., Suppl.*, 1908, 265—272).—The phosphorus-containing substances in foods fall into five groups, namely, inorganic phosphates, phosphatides, nuclein, phosphoproteins, and esters of phosphoric acid. The position of inosic acid and phosphoric acid in this classification is uncertain. A method is described for estimating these various compounds, and the results of the examination of various foods, meat, milk, bread, and other vegetable foods, are given in a table.

W. D. H.

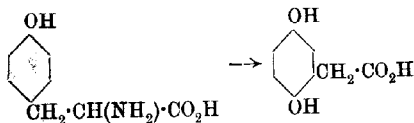
Absorption and Assimilation of Organic Compounds of Phosphorus. PIO MARFORI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 378—388).—Natural glycerophosphoric acid differs from the synthetic substance in that its salts, when injected subcutaneously, yield, at least in part, assimilable phosphorus. The phosphorus of lecithin, and, to some extent, that of nucleo-proteins, is also assimilated under these conditions. Nuclein and nucleic acid from yeast, when given by the mouth, did not increase phosphorus assimilation.

G. B.

The Cleavage of 2:5-Diketopiperazines in the Organism of the Rabbit. II. EMIL ABDERHALDEN and LEONHARD WACKER (*Zeitsch. physiol. Chem.*, 1908, 57, 325—328. Compare this vol., ii, 521).—In the further investigation of this question, it appeared desirable to employ an anhydride which is decomposed by alkali with difficulty. The one selected was *dl*-leucylglycine anhydride, and the experiments confirm those previously reported; this substance is in small measure resolved into its components by the organism of the rabbit.

W. D. H.

The Degradation of Aromatic Substances in the Human Organism. LEON BLUM (*Arch. exp. Path. Pharm.*, 1908, 59, 273—298).—When administered to normal individuals, neither phenylalanine nor tyrosine, normal hydrolysis products of proteins, increase appreciably the aromatic contents of the urine. On the other hand, when given to alcaptonurics, they increase the homogentisic acid excretion in the urine. It has been assumed, therefore, that homogentisic acid is a normal intermediate product of metabolism of aromatic hydrolysis products of proteins, and that alcaptonuric individuals do not possess the power of completely utilising these



products, and that tyrosine in the normal individual is first converted into homogentisic acid. This change can take place in the

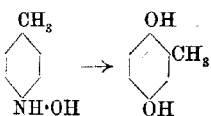
following ways: (a) the degradation may commence in the side-chain,

in which case *p*-hydroxyphenylacetic acid, or hydro-*p*-coumaric acid, $\text{OH}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, is first formed, and subsequently converted into homogentisic acid. It was found by Baumann and Schotten that hydro-*p*-coumaric acid, when administered to normal individuals, gave rise to *p*-hydroxybenzoic acid to the extent of 13%, whereas *p*-hydroxyphenylacetic acid was recovered to the extent of 78.6% in the urine. The author found that neither of these acids, on administration to alcaptonurics, caused an increase in the homogentisic acid output.

(b) A change in the relative positions of the hydroxyl group and the side-chain may take place with the formation of *m*- or *o*-tyrosine, which, on secondary oxidation in the para-position and a degradation of the side-chain, could give rise to homogentisic acid. It was found that neither of these tyrosines, when administered to alcaptonurics, increased the homogentisic acid output, and when given to normal individuals they were excreted in the form of the corresponding hydroxyphenylacetic acids to the extent of about 30%.

(c) The side-chain may be degraded to acetic acid, with a concurrent change in the relative position to the hydroxyl group, forming *m*- or *o*-hydroxyphenylacetic acid. Homogentisic acid would be formed from such products by subsequent oxidation in the para-position. This possibility was unlikely, as neither *o*- nor *m*-tyrosine gave rise to homogentisic acid, and it was also found that *m*-hydroxyphenylacetic acid, when administered to normal individuals, was found unchanged in the urine to the extent of 80%. Neither *o*- nor *m*-acid gave rise to increased homogentisic output in alcaptonurics.

(d) The processes of degradation, change of relative positions of the hydroxyl group to the side-chain containing the acid group, and of secondary oxidation, can take place concurrently. A similar change has been observed by Bamberger, who showed that tolylhydroxyl-



amine is converted by hot dilute sulphuric acid into toluquinol. Other examples of similar action are also known, and quoted by the author. A change of this description must take place when tyrosine is converted into homogentisic acid. The results also

indicate that homogentisic acid is a normal intermediate product of metabolism, as none of the products which were not fully destroyed in the normal individual gave rise to increased homogentisic output in alcaptonurics.

m-Tyrosine was prepared by the condensation of *m*-hydroxybenzaldehyde with hippuric acid. The lactimide of *m*-hydroxybenzoylaminocinnamic acid in the form of its acetyl derivative, $\text{C}_{16}\text{H}_{13}\text{O}_5\text{N}$, m. p. 149° , was obtained. On hydrolysis with 10% sodium hydroxide solution, *m*-hydroxybenzoylaminocinnamic acid, $\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}$, m. p. $205\text{--}206^\circ$, was formed, which, on reduction with sodium amalgam, yielded *m*-benzoyltyrosine, $\text{C}_{16}\text{H}_{15}\text{O}_4\text{N}$, m. p. 180° . From this, *m*-tyrosine, $\text{C}_9\text{H}_{11}\text{O}_3\text{N}$, m. p. $280\text{--}281^\circ$, was obtained by hydrolysis with 20% hydrochloric acid.

o-Tyrosine was obtained by a similar synthetic method, although all the intermediate products were not obtained pure. *o*-Benzoyltyrosine melts at 176° , and *o*-tyrosine at $249\text{--}250^\circ$.

S. B. S.

Are there Reducing Ferments in the Animal Body? ARTHUR HEFFTER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 253—260).—The reduction processes which occur in animal organs or their extracts, or in vegetable tissues, have been attributed to enzyme action. Boiling does not abolish the action; this and other considerations lead the author to the conclusion that "reductases" do not exist.

W. D. H.

Spectroscopic Properties of Yolk of Egg. LOUIS LEWIN, A. MIETHE, and E. STENGER (*Pflüger's Archiv*, 1908, 124, 585—590).—Attempts are being made to establish relationships between the colouring matters of egg-yolk and blood on account of the close morphological relationship between the blood and yolk. The first step in this direction has been the careful spectroscopic examination of the colouring matter of the yolk. The plates used for photographing the spectra were dyed with isocol. Solutions in water, acetone, alcohol, ether, chloroform, and benzene were examined. The solutions were found to follow Kundt's rule, for example, the absorption bands of the chloroform and benzene solutions were 5—10 μ nearer the red end of the spectrum than the corresponding bands of the other solutions.

There are three characteristic bands at 480, 453, and 427, and feebler bands at 400 and 378. No other yellow colouring matter gives the same absorption bands, and it is thus possible to detect adulterants of yellow of egg by spectroscopic measurements.

J. J. S.

Chemico-physical Investigations on the Crystalline Lens. FILIPPO BOTTAZZI and NOE SCALINCI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 305—316. Compare this vol., ii, 966).—The crystalline lens contains an electro-negative protein, *facoprotein*, which is only soluble in water in the form of acid protein in absence of alkali, or of alkali protein, which is soluble also in absence of salts. The essential material of the lenticular fibre is, in normal conditions, a hydrogel of liquid or gummy consistency. The results are mainly of physiological interest.

T. H. P.

Higher Fatty Acids in the Liver after Removal. JOHN B. LEATHES (*Arch. exp. Path. Pharm., Suppl.*, 1908, 327—336. Compare *Abstr.*, 1904, ii, 355).—A full account of a research previously published, with a description of further experiments on the same lines. The increase of fatty acids previously noted does not invariably occur, but no light can be at present thrown on the nature of the processes involved.

W. D. H.

The Formation of Uric Acid in the Liver of Birds. ERNST FRIEDMANN and H. MANDEL (*Arch. exp. Path. Pharm., Suppl.*, 1908, 199—207).—The experiments recorded were made by perfusing the surviving liver of the goose with various mixtures. If uric acid is added to the perfusion fluid, none is retained or destroyed by the liver. If urea and sodium lactate or malonate are added, there is no increase in uric acid formation. Uric acid formation in the bird's liver is therefore not the simple synthesis it has been considered to be.

W. D. H.

Action of Certain Gases on Autolysis. LUIGI BELLAZZI (*Zeitsch. physiol. Chem.*, 1908, 57, 389—394).—Carbon dioxide favours, and oxygen is indifferent, or feebly inhibitory, towards, autolysis of the liver. W. D. H.

Decomposition of Caffeine by Extract of Ox-Liver. Y. KOTAKE (*Zeitsch. physiol. Chem.*, 1908, 57, 378—381).—Extract of ox-liver decomposes caffeine into xanthine, hypoxanthine, *l*-methyl-xanthine, and paraxanthine. The removal of the methyl groups is prevented by boiling the extract, or by the use of such protoplasmic poisons as toluene or chloroform. The action is therefore attributed to a ferment. W. D. H.

Action of Drugs on the Mammalian Uterus. HAROLD J. FARDON (*Bio-Chem. J.*, 1908, 3, 405—411).—The investigation of a number of drugs shows that the reaction of the uterus is that of a plain muscular organ supplied by sympathetic nerves, of both inhibitory and augmentative nature. Pregnancy and nicotine alter the relative influence of the two sets of fibres. W. D. H.

Inosite [in Flesh]. FRANZ ROSENBERGER (*Zeitsch. physiol. Chem.*, 1908, 57, 464—467. Compare Abstr., 1908, ii, 873).—One factor in the presence or not of inosite, or of its precursor, inositol, in flesh appears to be the time of year, which, as is well known, also affects the quantity of glycogen. W. D. H.

Hydrolysis of Fish Muscle. THOMAS B. OSBORNE and FREDERICK W. HEYL (*Amer. J. Physiol.*, 1908, 23, 81—89).—The results are compared with those previously published in relation to chicken muscle in the following table, where the figures show percentages of cleavage products:

	Halibut muscle.	Chicken muscle.
Glycine	0.0	0.68
Alanine	?	2.28
Valine	0.79	?
Leucine	10.33	11.19
Proline	3.17	4.74
Phenylalanine	3.04	3.53
Aspartic acid.....	2.73	3.21
Glutamic acid	10.13	16.48
Serine	?	?
Tyrosine	2.39	2.16
Arginine	6.34	6.50
Histidine	0.55	0.47
Lysine	7.45	7.24
Ammonia	1.33	1.67
Tryptophan	present	present
Total	50.25	62.15

The most marked difference is seen to be in the percentage yield of glutamic acid. W. D. H.

Cholesterol in Ox-Bile. ERNST SALKOWSKI (*Zeitsch. physiol. Chem.*, 1908, 521—523).—Cholesterol can be easily prepared from the dried

bile of commerce. Previous saponification is unnecessary. The view is held that cholesterol occurs as such in bile, not as an ester.

W. D. H.

Quantitative Researches on the Exhalation of Alcohols. JULIUS POHL (*Arch. exp. Path. Pharm., Suppl.*, 1908, 427—434).—The result of the experiments recorded is that alcohols of higher boiling points (*tert.*-amyl alcohol; *isopropyl* alcohol) are exhaled with the breath to a greater degree than those of lower boiling point (ethyl alcohol; methyl alcohol). This unexpected result cannot yet be explained.

W. D. H.

The Distribution of Nitrogen amongst the Various Products in Human Urine. LOUIS C. MAILLARD (*Compt. rend.*, 1908, 147, 710—712).—The mixed urine from ten men of from 22—25 years of age was examined on six consecutive days. Of the total nitrogen excreted, 5.73% was in the form of ammonia, 81.29% in that of urea, 1.65% in that of purine compounds, 1.43% in that of uric acid, 0.22% in that of purine bases, and 11.15% in other forms of combination (creatinine, oxyproteic acids, urochrome, hippuric acid, amino-acids, etc.). The proportion of nitrogen to phosphorus was as 9:1.37. Muscular work was without appreciable influence on the total nitrogen excretion, although there was a slight diminution of urea, an undoubted increase in phosphates, and a slight increase in the nitrogen in the undetermined forms.

S. B. S.

Detection of Organic Bases in Urine. R. ENGELAND (*Zeitsch. physiol. Chem.*, 1908, 57, 49—64).—Urine has been treated by the three following methods: 1. Precipitation of the urine with a cold saturated solution of mercuric chloride and sodium acetate (compare Johnson, *Abstr.*, 1888, 506; 1889, 165). 2. Concentration of the urine and precipitation with tannin. 3. Precipitation of urine with a hot saturated solution of mercuric chloride and sodium acetate.

The bases isolated from the precipitate in the first method were creatinine and *as*-dimethylguanidine, the latter of which was obtained as the crystalline *aurichloride*, m. p. 144°.

The precipitate obtained according to the second method contained creatinine and methylguanidine, the latter of which was isolated as its *aurichloride*, m. p. 198°. It has been shown that the methyl- and dimethyl-guanidines are not produced by the action of hydrochloric acid on creatinine.

When the third method of precipitation was used, all the creatinine is removed, as the filtrate no longer gave Weyl's reaction. The bases isolated from the precipitate were creatinine, methylguanidine, vitiatine, histidine, a base, $C_{15}H_{26}O_{12}N_8$, similar to some of the protamines, and a base, $C_8H_7O_2N_3$, similar to histidine. The last base was isolated as its *picrolonate*, $C_8H_7O_2N_3 \cdot C_{10}H_8O_5N_4$, which crystallises in short needles, decomposing at 244°. The base gives a red coloration with alkali and copper sulphate, and also a dark red colour with an alkaline solution of diazobenzenesulphonic acid.

Iminazole derivatives are found in the urine of most animals, but

herbivorous animals produce larger quantities of these compounds than do the carnivora. J. J. S.

The Regular Occurrence of Indole in the Distillate of Normal Urine. MAX JAFFE (*Arch. exp. Path. Pharm., Suppl.*, 1908, 299—308).—Indole is constantly found in the urine of man and other animals examined. The methods of obtaining and identifying it are given in full, the differences in quantity in various animals noted, and the conditions in which indole derivatives occur discussed.

W. D. H.

Urinary Pigments derived from Indole. ALBERICO BENE-
DIGNI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 64—74. Compare
Abstr., 1907, ii, 980).—After subcutaneous administration, 5-methyl-
indole appears in the urine as dimethylindigotin; 2:5-dimethylindole
forms a red colouring matter, and α -naphthindole yields a reddish-
brown coloration, changing later to bluish-green. G. B.

Excretion of Urobilin in Disease. N. F. SURVEYOR (*Bio-Chem.*
J., 1908, 3, 439—448).—From an examination of 500 specimens of
urine in health and disease, the conclusion is drawn that there is no
correspondence between the amount of urobilin excreted and the
amount of ethereal sulphates in the urine. Urobilin formation is there-
fore not the result of intestinal putrefaction. Disease of the liver also
does not seem to be responsible for its appearance. The method
adopted for the estimation of urobilin is the depth of the absorption
band in an amyl-alcoholic extract of the urine, and so far as any con-
clusion can be drawn from the inconstant results found in most
diseases, it appears that conditions that lead to hæmoglobin destruc-
tion are those most likely to produce increase of the urinary
urobilin. W. D. H.

Microchemical Changes occurring in Appendicitis. OWEN T.
WILLIAMS (*Bio-Chem. J.*, 1908, 3, 391—401).—The author considers
that intestinal sand (Abstr., 1907, ii, 906) consists largely of calcium
salts (soaps) of saturated fatty acids. These soaps are not so easily
absorbed as those derived from unsaturated fatty acids.

Action of Radium Emanations [in Diabetes]. E. POULSSON
(*Arch. exp. Path. Pharm., Suppl.*, 1908, 443—448).—It is well known
that many mineral waters contain radium. Cases of diabetes were
treated with radon active water. In two severe cases, no good was
done; in a third case, where the disease was not so malignant, the
excretion of sugar was lessened, although whether this was due to the
water is uncertain. W. D. H.

The Degradation of Fatty Acids in Diabetes Mellitus.
JULIUS BAER and LEON BLUM (*Arch. exp. Path. Pharm.*, 1908, 59,
321—330).—It has been shown previously (compare Abstr., 1907, ii,
285) that isovaleric acid gives rise to β -hydroxybutyric acid in severe
cases of diabetes mellitus, and leucine acts in a similar way; no

great difference could be detected in the relative amounts excreted. Experiments were made in mild cases of the disease, and it was found that isovaleric acid exerted but little influence on the excretion of the hydroxybutyric acid; *n*-butyric and hexoic acids caused, however, an increased output. Similar results were not obtained in all the cases investigated. S. B. S.

The Influence of Muscular Work on the Excretion of Sugar in Pancreas Diabetes. Y. Seo (*Arch. exp. Path. Pharm.*, 1908, 59, 341—363).—Experiments were carried out on dogs which had been either wholly or partly deprived of the pancreas. The excretions of nitrogen and sugar were determined during periods of rest and periods of work, when the animals turned a treadmill. In animals in which the pancreas had only been partly removed, it was found that muscular work diminished the excretion of sugar. This was not the case, however, in animals on which total extirpation of the pancreas had been performed. In these cases, the ratio dextrose: nitrogen increased during the periods of work. The conclusion is drawn that increased utilisation of sugar by muscular work can only take place when some functioning pancreas tissue remains in the organism. S. B. S.

Acidosis in Pancreas Diabetes. EDUARD ALLARD (*Arch. exp. Path. Pharm.*, 1908, 59, 388—396).—Brugsch and others have maintained that acidosis is not found in cases of severe pancreas diabetes. The author cites, however, several experiments made with dogs deprived of the pancreas, in which a comparatively large output of β -hydroxybutyric acid was observed. The phenomenon of acidosis in these cases is, however, a very inconstant one, and it is suggested that it is due to secondary causes. The liver, or other parenchymatous tissue, in the advanced stages of the disease may have lost the power of degrading acetoacetic acid or acetone to simpler substances. S. B. S.

Radioactivity of Goitrogenic Springs. RÉPIN (*Compt. rend.*, 1908, 147, 703—705. Compare this vol., ii, 796).—The author has measured the radioactivity of the water of fourteen springs, a well, and several torrents situated in districts where goitre is endemic. The springs issuing from faults at the base of high mountains were all found to be radioactive, whilst surface water and torrents fed by glaciers or snow were inactive. Torrent waters when without turbidity are preferred, and the people who use them are free from goitre. A goitrous family living in a non-goitrous district was employing a radioactive well water. The prevalence of goitre in mountainous districts, and the success of distilled water in treating it, also suggests a connexion between radioactive water and the disease. The radioactivity has the character of radiothorium. R. J. C.

The Pathogenesis of Ochronosis. OSCAR GROSS and EDUARD ALLARET (*Arch. exp. Path. Pharm.*, 1908, 59, 384—387).—The colouring of cartilage in the pathological condition known as ochronosis

is due to the same anomaly in metabolism that produces alcaptonuria, namely, the production of homogentisic acid, which is not destroyed in the organism. The cartilage appears to have the property of attracting this acid and converting it into a dark pigment, producing in this way a form of arthritis, designated by the authors arthritis alcaptonurica. Almagia has shown that in cartilage soaked in solutions of sodium urate, concretions are formed similar to those found in cases of gout, and the authors show that cartilage in nearly neutral homogentisic acid solutions acquires a dark colour similar to that observed in cases of ochronosis. Other tissue does not act in this way.

S. B. S.

A Case of Chronic Pentosuria. RICCARDO LUZZATTO (*Arch. exp. Path. Pharm., Suppl.*, 1908, 366—377).—The urine of the patient (who has remained in good health for many years) contains generally 0.1% of *L*-arabinose. The amount is not affected by ingestion of large quantities of dextrose, sucrose, or starch, but is increased by galactose, by alkalis, and by intellectual work, and diminished by the ingestion of hydrochloric acid, and apparently also by excessive muscular labour. Pentosuria is therefore quite distinct from diabetes. In order to determine the reducing power of the urine, lead acetate is added, and, after filtration, ammonia; the basic lead precipitate formed carries down all the pentose, and is then redissolved in acid.

G. B.

Contents of a Cystic Tumour of the Breast. EMIL ZDAREK (*Zeitsch. physiol. Chem.*, 1908, 57, 461—463).—A complete analysis of the contents of a so-called butter-cyst of the mammary gland is given. It occurred in a woman, forty years of age, and had lasted twelve years before it was removed. It was about the size of an apple. It contained 48% of water, 38.6% of fat, 7.5% of fatty acids, 3% of coagulable protein, 1% of caseinogen, and 1.6% of ash.

W. D. H.

Action of Barium Chloride, Adrenaline, and Peptone on the Vaso-motor Apparatus. L. POPIELSKI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 435—442).—The three substances named all act on the peripheral vaso-motor mechanism. Barium chloride and adrenaline cause a rise of pressure by acting on the musculature; Witte's peptone causes a fall by acting on the nerve-endings in the vessels. The substance responsible for the activity of Witte's peptone is named *vaso-dilatin*, it is not a protein, nor is it choline, which when pure produces the opposite effect. Vaso-dilatin is also the substance which renders the blood incoagulable.

W. D. H.

Action of Certain Narcotics on Nerve. ALBRECHT BETHE (*Arch. exp. Path. Pharm., Suppl.*, 1908, 75—82).—The excitability of nerve and its reaction to the constant current (polarisation picture) go hand in hand. Solutions of chloral hydrate, ethylurethane, and henylurethane, which abolish the excitability, also abolish the normal polarisation changes. If the dose of the narcotic is insufficient to completely abolish excitability, the polarisation changes are altered in the measure. When these changes are produced, immersion in

Locke's solution causes the nerve to recover. This reversibility is least easy to obtain after chloral hydrate. W. D. H.

Pharmacological Action of Certain Lactones and the Corresponding Hydroxy-acids. CHARLES R. MARSHALL (*Arch. exp. Path. Pharm., Suppl.*, 1908, 389—394. Compare Abstr., 1906, ii, 788).—Certain tropeines containing a lactone group (Jowett and Hann, *Trans.*, 1906, 89, 357; Jowett and Pyman, *Trans.*, 1907, 91, 93), and possessing an atropine-like action, lose this action when they are converted into salts of the corresponding hydroxy-acids. After the addition of a molecular quantity of alkali hydroxide, this conversion occurs relatively slowly towards the end of the reaction, and the gradual change can be demonstrated pharmacologically. G. B.

Behaviour of Sodium Salicylate in the Organism. ALESSANDRO BALDONI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 54—63).—Sodium salicylate, when administered to dogs, is mostly excreted unchanged in the urine, but a small portion is converted into two crystalline derivatives with acid properties, both yielding a blue coloration with ferric chloride and having a strong reducing action. Of these, one, m. p. 169—170°, has the composition $C_{16}H_{16}O_8N$; the other, m. p. 187—188°, has the composition $C_{16}H_{14}O_8$. Salicylic acid, which is found in human urine after ingestion of sodium salicylate, cannot be detected in dogs' urine. G. B.

Pharmacological Significance of Twin Ethyl Groups. SIGMUND FRÄNKEL (*Arch. exp. Path. Pharm., Suppl.*, 1908, 181—187).—In order to test the hypothesis, that the hypnotic action of such drugs as veronal (diethylbarbituric acid, $CO < \begin{smallmatrix} NH \cdot CO \\ NH \cdot CO \end{smallmatrix} > C_2Et_2$) is due to two ethyl groups attached to the same carbon atom of the ring, the author has examined tetra-, penta-, and hexa-ethylphloroglucinol, and also the hexamethyl derivative. None of these substances has a hypnotic action, but they produce strychnine-like convulsions. G. B.

Pharmaco-dynamic Characters of Coumarin. ALEXANDER ELLINGER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 150—163).—The toxic action of cantharidin on the kidneys suggested the examination of other substances with a lactone structure, and coumarin was selected. In rabbits, albuminuria may occur, but only to any extent when the urine is acid. The injury to the kidneys, even in large doses, is never serious. Coumarin, however, in frogs causes deep narcosis, owing to its action on brain and cord; the centres affected include those governing cardiac inhibition and respiration, but the vaso-motor centre is not affected. In rabbits it is, in suitable doses, a harmless narcotic, and has no effect on the heart; large doses injected intravenously paralyse the respiration and produce death. In dogs, also, it is a narcotic, and usually produces vomiting. In large doses it kills them, but without the characteristic symptoms noticed in rabbits. This difference in action is probably related to a difference in

excretion in the two animals, but this part of the work is still in progress.
W. D. H.

Behaviour of Atropine in Various Animals. MAX CLOETTA (*Arch. exp. Path. Pharm., Suppl.*, 1908, 119—125).—Atropine appears to be destroyed in the brain and liver; this is most marked in the rabbit's brain, and least in the cat. This appears to be related to what is termed the "vital reaction difference" of the nervous tissues of various animals, and is not yet explicable on a chemical or physical basis.
W. D. H.

Action of Caffeine on Frogs. CARL JACOB and GOLOWINSKI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 286—298).—The relative difference in the action of caffeine on the two species of frog (*Rana esculenta* and *R. temporaria*) has been attributed to differences in the excitability of the spinal cord. The present research deals mainly with the difference between the muscles of the two species. The difference is mainly one of elasticity and extensibility, and this, as well as the different behaviour of caffeine towards them appears to depend on the sarcolemma of the muscular fibres, and the amount or character of its lipoids.
W. D. H.

The Fate of Synthetic Muscarine in the Animal Body. HERMANN FÜHNER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 208—213).—If muscarine is given subcutaneously to tortoises, it is again obtainable in the urine during the next few days in active form, and but little, if at all, altered in amount. The same is true for curarine.
W. D. H.

Poisons of Amanita Phalloides. JOHN J. ABEL and WILLIAM W. FORD (*Arch. exp. Path. Pharm., Suppl.*, 1908, 8—15. Compare Abstr., 1907, ii, 192).—Further details are given of the properties and actions of the two poisons contained in this fungus, namely, the hemolysin, which is a glucoside containing pentose, and the toxin. The hemolysin is easily destroyed by the gastric juice, so that, if taken by the stomach, it does not manifest its hemolytic properties.
W. D. H.

Action of Atoxyl on the Animal Body. J. IGERSHIMER (*Arch. exp. Path. Pharm., Suppl.*, 1908, 282—285).—Although sodium *p*-aminophenylarsinic acid may have its proper sphere of action in dealing with diseases due to protozoa (trypanosomiasis, syphilis, etc.), it should be recognised that its other name, *atoxyl*, is an illusory one, and in man it produces effects on the nervous, excretory, and alimentary system. Details are given of its toxic action on a number of dogs and cats.
W. D. H.

Action of Poisons on Enzymatic Processes. KARL G. SANTESSON (*Arch. exp. Path. Pharm., Suppl.*, 1908, 469—481).—It is suggested that the harmful influence of such poisons as hydrocyanic acid on the heart and on plant life is due to interference with intra-

cellular enzymes, the action of which forms the basis of the activity of living cells. In support of this view, experiments are recorded with muscle extract, and the action of the catalase in it was measured by the evolution of gas which occurs when mixed with hydrogen peroxide. This action is favoured by dilute alkali, hindered by dilute acid, by hydrocyanic acid, and also by manganese sulphate in decinormal solution; but in concentration $N/1000$ the development of gas is slightly increased; the favouring action of dilute solutions of manganese salts on other enzymatic processes is well known.

W. D. H.

Poisoning with Potassium Chlorate. L. RIESS (*Arch. exp. Path. Pharm., Suppl.*, 1908, 460—468).—The hæmolysis which follows poisoning with potassium chlorate is of a specially pernicious character, and differs from other cases of hæmolysis in that the corpuscles are broken up, so that irregular clumps of hæmoglobin are formed. The urinary tubules become filled with similar particles, although how they pass the renal epithelium is a difficulty; these tubules, especially in chronic cases, get filled with these particles, which blend so as to form casts of the tubules.

W. D. H.

Post-mortem Action of Corrosive Poisons in the Stomach. ERICH HARNACK and HERMANN HILDEBRANDT (*Arch. exp. Path. Pharm., Suppl.*, 1908, 246—252).—In forensic medicine, the degree of action of a caustic poison after death is sometimes raised. It is shown by experiments on cats that their destructive action on the gastric mucous membrane is more marked after death than during life. The action of potassium cyanide on the blood pigment also occurs post-mortem, but absorption of ammonia from the stomach only takes place during life.

W. D. H.

Chronic Oleic Acid Poisoning. EDWIN S. FAUST (*Arch. exp. Path. Pharm., Suppl.*, 1908, 171—175).—The idea that the anæmia in those afflicted with the tape-worm, *Bothriocephalus*, is due to oleic acid contained as a cholesterol ester in the worm acting as a hæmolytic poison, led to the present research, in which it is shown by experiments on rabbits and dogs that oleic acid administered by the mouth or subcutaneously over long periods of time does act as a hæmolytic agent in the same way as it does *in vitro*; the red corpuscles are diminished in number, and the hæmoglobin is lessened in amount.

W. D. H.

So-called Antitoxic Power of Animal Tissues towards Strychnine. PAUL PELLACANI and FOLLI (*Arch. exp. Path. Pharm., Suppl.*, 1908, 419—426).—Proofs are adduced that the tissues do not possess the power of destroying strychnine; practically the whole of it (98%) can be recovered from the tissues many hours after its introduction.

W. D. H.

Chemistry of Vegetable Physiology and Agriculture.

Estimation of the Reducing Power of Bacteria and Animal Organs. HEINRICH WICHERN (*Zeitsch. physiol. Chem.*, 1908, 57, 365—377).—An important distinction between different micro-organisms is their relative reducing powers, and most methods hitherto employed are defective from the quantitative aspect. It is, however, possible to obtain good results with certain coloured substances, the colour of which disappears on reduction; of these, methylene-blue appears to be the best. Still better results are obtained with ferric chloride and titration with titanium trichloride. This is illustrated by the experiments recorded with various bacteria. The method as applied to pieces of organs is not so useful, although fairly concordant results are obtained with extracts of organs. It is suggested that the same method might be employed for the estimation of the activity of oxydases.

W. D. H.

Bacterial Formation of Sulphates in Sewage Purification. CH. ROUCHY (*J. Pharm. Chim.*, 1908, [vi], 28, 439—444).—The opalescence or milkiness of effluents from inefficient bacterial sewage beds is due to the presence of finely-divided sulphur. In the limpid effluents from beds which are working properly, the sulphur has been entirely converted into sulphuric acid, which, reacting on the carbonates in the sewage, is converted into sulphates. This formation of sulphuric acid is, the author thinks, due to the oxidising action of special bacteria, analogous to the nitrifying organisms.

T. A. H.

Oxidation by means of Moulds. REGINALD O. HERZOG and A. MEIER (*Zeitsch. physiol. Chem.*, 1908, 57, 35—42. Compare Harden, *Trans.*, 1903, 83, 424).—Cultures of *Penicillium glaucum* were grown in dilute beer wort, and when the evolution of carbon dioxide had reached a constant value, a solution of the ammonium salt of a hydroxy-acid was added. The evolution of carbon dioxide was increased considerably, and each experiment was continued until the evolution had fallen again to the normal value for the medium. In this way, the carbon dioxide due to the decomposition of the hydroxy-acid could be calculated. The following acids were readily attacked: lactic, tartaric, malic, mandelic, β -hydroxybutyric, and in every case the amount of carbon dioxide evolved was considerably in excess of that calculated for the amount of acid destroyed.

Glycollic, citric, pyruvic, and hydroxyisobutyric acids were not affected.

The conclusion is drawn that the process is one of oxidation, but is accompanied by another unknown reaction, which also gives rise to carbon dioxide.

It has been shown that, when the organism has been destroyed by acetone or methyl alcohol, it can still decompose the hydroxy-acids, thus indicating that the reaction is due to an oxidising enzyme. The

activity of the dead cells is not so marked as that of the living, and ceases after some thirty-six hours. J. J. S.

Conversion of Cinnamic Acid into Styrene by means of Moulds. REGINALD O. HERZOG and O. RIPKE (*Zeitsch. physiol. Chem.*, 1908, 57, 43—45. Compare Oliviero, *Abstr.*, 1906, ii, 623).—Styrene is formed when *Aspergillus niger* is grown in dilute beer wort containing ammonium cinnamate (0.25% solution). The amount of hydrocarbon formed can be determined by aspirating sterilised air through the liquid, and then passing it through ten bulbs containing carbon disulphide. The styrene is weighed as its dibromide.

Attention is drawn to the importance of this type of reaction for the explanation of the formation of mineral oil deposits. J. J. S.

Transformations of the Chromogenic Matter of Grapes during Maturation. J. LABORDE (*Compt. rend.*, 1908, 147, 753—755).—It has been shown previously (this vol., ii, 774) that the colouring matter of red grapes can be artificially produced from the œnotannin of the unripe grapes. The object of the present work is to discover how the transformation is effected naturally. The tannins exist in the green pellicles in two forms: (1) soluble in strong alcohol, (2) insoluble in this solvent, the latter being the greater in amount. By determination of the amounts of these tannins in various species of red and white grapes, in varying states of maturity, by means of a colorimetric method described, it is shown that the total quantity of tannin matter diminishes, the proportion of the soluble tannin increases, and that of the insoluble tannin diminishes, during ripening. It is during this change of the insoluble into soluble tannin that the colouring matter of the red grapes appears. Colorimetric examination of the hydrochloric acid solution from the pellicles of grapes just commencing to ripen, before and after heating in an autoclave, shows that they contain untransformed œnotannin; with the ripe grapes this is not observed. The œnotannin in the wine must therefore proceed from other solid parts. The soluble tannin obtained from the pellicles of ripe white grapes gives only an insoluble, brown colouring matter when heated with 2% hydrochloric acid in an autoclave. The change in solubility of the tannin is probably due to diastatic action, and this raises the question whether it is an enzyme which transforms the œnotannin into the colouring matter of red grapes; if so, this enzyme must be absent from the white grapes. All attempts to find such an enzyme have, however, proved unsuccessful. E. H.

Carbohydrates of Coelococcus and Phytelephas. SERGIUS IVANOFF (*J. Landw.*, 1908, 56, 217—228).—Ground *Coelococcus* shavings, when hydrolysed with 6% sulphuric acid, yielded 20% of mannose; *Phytelephas macrocarpa* gave 37%. No other hexose was produced in appreciable quantity. Both substances yielded arabinose when boiled with 2—3% sulphuric acid; xylose could not be detected. Mannose was found to be present in two modifications, as hemicellulose and as mannocellulose. N. H. J. M.

Indole in Flowers. F. WEEHUIZEN (*Pharm. Weekblad*, 1908, 45, 1325—1329).—Indole can be detected in the white flowers of *Murraya exotica* by the action of vanillin or *p*-dimethylaminobenzaldehyde on the alcoholic extract in presence of concentrated hydrochloric acid. Each reagent produces a red coloration, that with *p*-dimethylaminobenzaldehyde being more intense in presence of sodium nitrite. Since phloroglucinol answers to the same test when it is present, the vapour exhaled from the flowers should be allowed to come into contact with the reagents.

A. J. W.

Abnormal Biochemical Products of the Rue Anemone. FREDERICK S. BEATTIE (*Amer. Chem. J.*, 1908, 40, 415—428).—Fasciated specimens of rue anemone (*Syndesmon thalictroides*) contain about 20% of methyl and ethyl isocarbostyryl-3-carboxylates and 3-methylquinoline-4-carboxylic acid. These substances are not found in the normal plant.

J. C. C.

Preparation of Pure Chitin from Boletus edulis. EMIL SCHOLL (*Monatsh.*, 1908, 29, 1023—1036).—The membranes of *Boletus edulis* consist chiefly of chitin in loose combination with carbohydrates. It has been found possible to prepare pure chitin from this fungus to the extent of 5 to 6% of the dried plant by alternate treatment with boiling water and boiling 10% aqueous potassium hydroxide. The chitin so obtained is chemically identical with animal chitin, and is, unlike Winterstein's fungus-cellulose (*Abstr.*, 1896, ii, 210), completely insoluble in concentrated alkalis. It yields about 78% of crystalline glucosamine hydrochloride when hydrolysed with hydrochloric acid.

W. H. G.

Peptolytic Ferments in Germinating and Ungerminated Seeds of Various Plants. EMIL AEDERHALDEN and DAMMHAHN (*Zeitsch. physiol. Chem.*, 1908, 57, 332—338).—The existence of peptolytic enzymes in plants has been established by the work of Schulze and Winterstein. Schittenhelm found also peptolytic ferments in seeds, and this observation is confirmed; extracts of the seeds of wheat, maize, barley, and lupins produce splitting of glycyl-*L*-tyrosine if the seeds have germinated. In the resting stage, peptolytic ferments are absent.

W. D. H.

Assimilation and Elimination of Nutrients by Oats at Different Periods of Vegetation. L. SEIDLER and ALBERT STUTZER (*J. Landw.*, 1908, 56, 273—278).—Pot experiments with oats in two different soils (a heavy loam mixed with gravel and a chalky gravel mixed with peat), manured with superphosphate and sodium nitrate and with varying amounts of potassium chloride. The plants were analysed (1) four weeks after sowing the seed, (2) when the ears began to form, (3) at the period of full flower, and (4) when the ripening was complete.

As regards nitrogen, it was found that from 50 to 60% of the total amount assimilated was taken up in the first four weeks, and that the ripe plants contained from 6 to 26% less nitrogen than at the third

period. The highest amounts of potassium were also found in the third period, the losses during ripening increasing, with some exceptions, with the amount of potassium chloride applied.

Sodium, calcium, and phosphoric acid seem to be retained by the plants to the end; slight losses of calcium occasionally took place between the third and fourth periods.

N. H. J. M.

Influence of Different Manurial Conditions on the Assimilation of Nutrients and the Structure of Plants. MAX WAGNER (*Landw. Versuchs-Stat.*, 1908, 69, 161—233).—Two series of pot experiments in which mustard, buckwheat, barley, and oats were grown under different manurial conditions. In the second series, plants were taken up and analysed at four periods of growth (May 9 and 29, June 25 and July 13).

As regards the final amount of growth above ground, both the oats and the barley, which ripened completely, generally showed a loss, especially when insufficiently manured. Deficiency of nutrients, especially nitrogen, resulted in a higher relation of roots to above ground growth.

A deficiency of phosphoric acid in soil otherwise sufficiently manured reduced the yield of barley most, then oats, buckwheat, and mustard. When potassium was deficient, barley again suffered most, then oats and mustard, and lastly buckwheat.

Whilst the buckwheat, which remained green to the fourth period, continued to take up the different nutrients to the end, the oats, barley, and mustard showed losses of nitrogen, potassium, and phosphoric acid at the final period.

The relation of grain to straw was considerably affected by the conditions of manuring. In the case of barley, a low relation of grain was most marked when potassium was deficient, whilst with oats, deficiency of nitrogen caused the greatest reduction.

N. H. J. M.

Amount of Nutrients Utilised by Sugar-Beet in the First Year and its Relation to the Amount of Sugar in the Roots. KARL ANDERLIK and JOSEF URBAN (*Zeitsch. Zuckerind. Böhm.*, 1908, 33, 83—94. Compare *ibid.*, 1906, 31, 149; 1907, 32, 559).—The amount of phosphoric acid assimilated by sugar-beet depends on the manuring and the amount present in the soil, on the rainfall, and on the seed. With a yield of 400 quintals of roots, the amount of phosphoric acid taken up under normal conditions varied from 51.7 to 87.8 kilos., whilst in a dry season the average amount was 48.6 kilos.

The amount of phosphoric acid required to produce 100 parts of sugar also varies according to manurial and climatic conditions. The results of various experiments made from 1902 to 1905 showed that the average amount is 0.97 part of P_2O_5 , the amount deposited in the roots being 48.4% of the total.

N. H. J. M.

Digestibility of Hay from Water Meadows as Compared with Ordinary Hay. KONRAD FRIEDLÄNDER (*Landw. Versuchs-Stat.*, 1908, 69, 245—258).—The hay from water meadows is characterised

by a high percentage of mineral matters and a very high percentage of crude protein. The results of feeding experiments with sheep showed that the digestibility of the crude protein is distinctly higher, and of the true protein appreciably higher, than in the case of ordinary hay. The latter contains, however, a higher amount of carbohydrates.

N. H. J. M.

Studies on the Soils of the Northern Portion of the Great Plains Region: Nitrogen and Humus. FREDERICK J. ALWAY and ROBERT S. TRUMBULL (*Amer. Chem. J.*, 1908, 40, 147—149. Compare Abstr., 1907, ii, 294).—Determinations of total nitrogen, soluble humus, and the nitrogen in the soluble humus in nineteen comparatively heavy soils and one sandy soil from Saskatchewan and Alberta. The percentages of total nitrogen are fairly high in all the soils except the sand, whilst the humus is rather low, although much higher than in the arid soils of California. The proportion of the total nitrogen present in the form of humus is decidedly low. The percentage of nitrogen in the humus is not markedly different from that of soils from humid regions.

The results seem to indicate that the surface soils of the semi-arid portions of Western Canada have the characteristics of humid regions, whilst the sub-soils show the peculiarities of other arid regions.

N. H. J. M.

Isolation of Dihydroxystearic Acid from Soils. OSWALD SCHREINER and EDMUND C. SHOREY (*J. Amer. Chem. Soc.*, 1908, 30, 1599—1607. Compare this vol., ii, 889).—Four out of six more or less unfertile soils were found to contain dihydroxystearic acid, identical with the one obtained from elaidic acid (Saytzeff, Abstr., 1886, 140). The soil which yielded most of the substance was a grey silt loam from Tennessee, which had been under cultivation for more than fifteen years chiefly in cotton. The soil contains organic matter 3.26, and nitrogen 0.16%. The amount of fairly pure substance obtained from 1 kilo. of soil was about 0.05 gram; the amount actually present is probably far greater.

As regards the toxicity of dihydroxystearic acid, experiments with wheat seedlings show that as little as 20 parts per million is distinctly injurious; 100 parts per million reduced the weight to 53%, whilst 200 parts per million (approximately a saturated solution) had about the same effect. Practically the same results were obtained with dihydroxystearic acid, prepared from elaidic acid, and with the isomeric acid from oleic acid, the weight being reduced to about half of that in the control experiment, and the plants being killed in twelve to fifteen days.

The Takoma soil previously examined (*loc. cit.*), which also contains dihydroxystearic acid, seems to be a good medium for fungi. Rootlets of oak trees growing in the soil were found to be infested with mould, and when freed from soil yielded small quantities of the acid. The conclusion is drawn that the dihydroxystearic acid is produced by the moulds, perhaps by the decomposition of lecithins

into oleic acid, conversion of oleic acid by nitrous acid into elaidic acid, and oxidation of the latter by enzymes or micro-organisms.

N. H. J. M.

Ammonia Question. PAUL EHRENBURG (*Landw. Versuchs-Stat.* 1908, 69, 259—294. Compare this vol., ii, 60).—Experiments on the production of basic and acid reactions in peaty soil, by application of nitrogen in the form of potassium or sodium nitrate and ammonium salt respectively, and on the effect of the reactions on different plants. Experiments were also made in which ammonium sulphate was applied both with and without calcium carbonate.

As an example of the effect of the two forms of nitrogen (in absence of calcium carbonate) on the growth of plants in an acid soil, it is shown that maize, sorghum, barley, and white mustard during the first periods of growth cause the acidity of the soil to be neutralised when manured with nitrate, and then produce normal growth. At the same time, the assimilation of potassium and other bases tends to maintain the change of reaction within certain limits. The same plants, manured with ammonium sulphate, were able, with the exception of mustard, to make a start under the conditions of slight soil acidity, but after reaching a certain point fell off, owing to the increased acidity resulting from the sulphuric acid of the ammonium salt. Mustard is the most sensitive to ammonium salts, and maize the least sensitive, owing probably to the greater amount of soil it produces.

N. H. J. M.

Old and New Nitrogenous Fertilisers: Calcium Cyanamide, Calcium Nitrate, Ammonia Sulphate, and Sodium Nitrate. VITTORIO NAZARI (*Atti R. Accad. Lincei*, 1908, [v], 17, ii, 334—342).—The author has investigated the influence of various factors on the fertilising value of calcium cyanamide, and has carried out experiments on the comparative values of calcium cyanamide, calcium nitrate, ammonium sulphate, and sodium nitrate as fertilisers. The crop used, in all cases, was wheat.

The results show that the value of calcium cyanamide is greatly enhanced by the presence in the soil of organic matter in the form of stable manure. The cyanamide acts most beneficially at about 20 cm. below the surface of the soil, and at a depth of 5 cm. gives much less favourable results than at a depth of 35 cm. The best fertiliser to use in association with calcium cyanamide is bone superphosphate, partly owing to the fact that it contains a certain proportion of readily fermentable matter, which serves to nourish the micro-organisms; also, admixture of the cyanamide with gypsum gives better results than admixture with lime, owing to the action of the calcium sulphate on the soil constituents containing insoluble potassium compounds, and to its stimulating action on many of the lower forms of plant life. On treating the soil with equal amounts of nitrogen in the form of the various fertilisers, the best and approximately identical results were obtained with calcium nitrate, ammonium sulphate, and sodium nitrate, calcium cyanamide yielding a considerably inferior crop.

T. H. P.

Danger of Employing Salts of Arsenic in Agriculture. W. MESTREZAT (*J. Pharm. Chim.*, 1908, [vi], 28, 393—397).—Wines manufactured from untreated vines are found to contain 0.000005 to 0.000008 gram of arsenic per litre, whilst those from vines which have been cultivated on the same soil and in the same manner as the preceding, except that they have been treated three times with various arsenical preparations (as insecticides), contain 0.000020 to 0.000025 gram of arsenic per litre. The danger from this cause is accordingly negligible. Inappreciable quantities of arsenic are found to be inhaled by workmen who have to prepare the arsenic salts for use, whilst the quantities settling on their hands and faces (0.0007 to 0.0012 gram) are probably innocuous. E. H.

Analytical Chemistry.

New Burette Clamp. GUSTAV MÜLLER (*Zeitsch. angew. Chem.*, 1908, 21, 2318—2319).—To a retort stand with a tripod base are fixed one or more clamps so constructed that they may be moved and turned both horizontally and vertically. Hence it is immaterial whether the stand is perfectly level, for the burettes may be always properly adjusted.

A retort stand with the rod fixed in the centre of the oblong base may also be used; the rod should consist of two parts, which may be unscrewed if desired. The base is provided with a hole, so that it may be attached to the wall; the clamps are then fixed to the protruding rod. From the rod may be suspended a variety of laboratory sundries, such as towels, brushes, etc., so as to economise space.

L. DE K.

Rapid Method of Qualitative Analysis. W. BRANCH POLLARD (*Chem. News*, 1908, 98, 211).—One part of the finely-powdered substance is mixed with 1 part of vaseline and 5 parts of sodium peroxide. The mixture is placed on a thick iron plate, or in the cavity of a scorifying mould, and ignited by means of a match or a Bunsen burner. The fused mass is extracted with water, and both the soluble and insoluble matter tested as usual. The soluble portion contains, in the highest state of oxidation, those elements which form soluble sodium salts; the insoluble portion contains the oxides and carbonates of the other metallic elements.

A special test should be made for mercury and sodium. The method is more particularly suited for examining ores and minerals in the field.

L. DE K.

Detection of Hydrogen Peroxide in Milk. W. PERCY WILKINSON and ERNST R. C. PETERS (*Zeitsch. Natur. Genussm.*, 1908, 16, 515—517).—The reaction described by Feder (*Abstr.*, 1908, ii, 318) is shown by

the authors' experiments to depend on the actual quantities of hydrogen peroxide and formaldehyde present, and also on the proportion of these two substances to each other. The strongest reaction is obtained when from 0.004 to 0.013% of formaldehyde and about 0.005% of hydrogen peroxide are present in the milk. If the amount of hydrogen peroxide is increased to 0.5%, a coloration is not obtained. Ferric salts, nitrates, etc., also influence the reaction, so that a positive reaction is not a definite proof of the presence of hydrogen peroxide. The test proposed by the authors (Abstr., 1908, ii, 907) for distinguishing between raw and heated milk may be applied conversely to the detection of hydrogen peroxide; it is not affected by the presence of nitrates or ferric salts.

W. P. S.

Pringsheim's Method for Estimating Chlorine, Bromine, and Iodine in Organic Compounds. ERIK J. VIRGIN (*Arkiv Kem. Min. Geol.*, 1908, 3, No. 12, 1—6).—The author has investigated the method proposed by Pringsheim (compare Abstr., 1904, ii, 146, 447; 1905, ii, 609) for estimating halogens in organic compounds by means of sodium peroxide.

With 1:4-dichloronaphthalene tetrachloride, 3:4-dichlorophenol, and *p*-bromoaniline, this method gives low results, whilst with *p*-dibromobenzene and iodoform good results are obtained.

The method is only applicable to substances which burn quietly with the sodium peroxide, and give no smoke or flame outside the crucible. As it is impossible to tell beforehand if this will be the case, and as there is no definite limit between quiet and vigorous combustion, the method is not to be recommended.

T. H. P.

Kjeldahl's Method. ERNST SALKOWSKI (*Zeitsch. physiol. Chem.*, 1908, 57, 523—526).—Some practical details in the employment of this method are given as the result of the author's experience. Among other points, the omission of the addition of mercuric oxide is recommended.

W. D. H.

Micro-chemical Reactions of Arsenic Applicable to Medico-Legal Investigations. GEORGES DENIGÈS (*Compt. rend.*, 1908, 147, 596—597).—A description of the technique is given for identifying arsenic by microscopical methods, the reagents employed being silver nitrate in solutions acidified by acetic acid, and in ammoniacal solutions. The reactions are carried out with a drop of arsenical liquid, which has been evaporated to dryness with certain precautions, on an object glass.

S. B. S.

Mercurous Nitrate as a Microchemical Reagent for Arsenic. GEORGES DENIGÈS (*Compt. rend.*, 1908, 147, 744—745. Compare preceding abstract).—The reagent is prepared by triturating crystallised mercurous nitrate (10 grams) with nitric acid (D 1.39, 10 c.c.) and adding water (100 c.c.). A small drop of the solution (in nitric acid) to be tested is evaporated to dryness on a glass plate by a gentle heat, and the residue treated with a drop of ammonia, which is also evaporated. To the residue, when quite cold, a drop of the mercurous reagent of a volume insufficient to completely cover it is added.

After two minutes, the drop of reagent is spread over the entire residue by means of a very finely-pointed glass rod, care being taken not to scratch the glass and to keep the rod in a continuous circular motion for 20—30 turns. After another two minutes, if arsenic is present, examination under the microscope (magnifying 40—50 or 100 diameters) reveals thick macles and crystallites, often arranged in a double fan-shape and coloured brownish-yellow, and groups of almost colourless tablets with rounded ends. When the residue is very small, only an extremely small drop (not more than 1—2 mm. diameter) must be used. The test can only be effected successfully by the exact attention to all the above details.

E. H. *

Simplified Apparatus for the Estimation of Carbon in Iron. THEO. GRAZESCHUK (*Chem. Zeit.*, 1908, 32, 1092).—An improvement of the apparatus generally used. Close to the end of the inner tube of the condensing arrangement is sealed a concave disk, on which is placed the sample, and when the condenser is placed in the flask the disk should dip slightly into the chromic acid mixture. After transmitting a current of purified air and connecting the apparatus with the train of absorbers, heat is applied with a small flame, and, owing to the disc, a more even distribution of heat is effected. Instead of using a breakable glass tube for connecting the condenser with the water supply, an indiarubber tube is substituted. The distance between the condenser and flask is about 2 mm. The acid rises to a considerable height in the tube, but there is no danger of loss.

L. DE K.

A Boat Funnel. H. STOLTZENBERG (*Zeitsch. angew. Chem.*, 1908, 21, 2271).—To facilitate filling the boat used for combustions, the author describes a nickel funnel made the shape and length of the boat and having a narrow slit underneath. This funnel is supported over the boat, which stands on a small nickel tray, and it allows of the substance being evenly distributed along the boat in an expeditious manner; if overfilled, the substance may be collected from the nickel tray.

J. V. E.

Apparatus for the Estimation of Carbon Dioxide, etc. M. EMMANUEL POZZI-ESCOT (*Bull. Assoc. chim. Sucr. Dist.*, 1908, 26, 267—271).—The piece of apparatus described consists of a flat-bottomed flask provided with a hollow glass stopper, through the centre of which is fused the stem of a tapped funnel; the latter serves as a reservoir for the dilute acid, or other reagent, used in the estimation. A narrow glass tube extends from the stopper of the bottle to the top of the funnel, and a second tube leading from the stopper serves as the delivery tube of the apparatus. The top of the funnel is closed by means of a stopper provided with a small hole, which, on turning the stopper, is brought opposite a similar hole in the neck, so that the pressure in the flask may be equalised when necessary. The apparatus may be used for the estimation of carbon dioxide in carbonates, nitrogen in urea and ammonia (by using the sodium hypobromite method), and in the analysis of hydrogen peroxide, persulphates, etc.

W. P. S.

Separation of the Alkali Metals in the Electrolytic Way. JACOB S. GOLDBAUM and EDGAR F. SMITH (*J. Amer. Chem. Soc.*, 1908, 30, 1705—1711).—In earlier papers (Abstr., 1907, ii, 574, 988), it has been shown that halide salts of various metals, and particularly those of the alkalis and alkaline earths, can be readily analysed electrolytically with the aid of a mercury cathode and a rotating silver anode. Results are now recorded which have been obtained with ammonium chloride, bromide, and thiocyanate, and the chlorides of caesium, rubidium, lithium, and which prove the accuracy of the method.

* An account is given of experiments on the separation of the alkali metals. Freudenberg (Abstr., 1893, ii, 506) has shown that trustworthy separations of metals may be obtained by arranging the pressure so that it exceeds the polarisation value of one metal and continues below that of the other. Working on this principle, separations of sodium from potassium, ammonium, caesium, rubidium, and lithium, of potassium from rubidium, caesium, and lithium, of caesium from rubidium, and of lithium from rubidium and caesium, have been successfully effected. The decomposition values of potassium and ammonium salts are so near to one another that these elements could not be separated.

It is suggested that this method of separation may prove useful in the estimation of small quantities of the alkali metals which occur in silicates. E. G.

Volumetric Alkalimetric Method for Determining Alkaline Earths in Manures and Soils. OTTO FOERSTER (*Landw. Versuchs-Stat.*, 1908, 69, 235—243).—The substance (4—5 grams of quicklime or 8—10 grams of carbonate) is heated with *N*-hydrochloric acid (200—250 c.c.) in a 400 or 500 c.c. measuring flask for half an hour, then filled to the mark, and the whole filtered. A portion of the filtrate (100 c.c.), after adding the indicator, is treated with *N*/2 sodium hydroxide until the colour changes, then with 1—2 c.c. of *N*/2 acid, and boiled for a few minutes. When there is no, or only slight, precipitation of sesquioxides, the solution may be at once titrated back with *N*/2 alkali, and the number of c.c. used added to the number previously obtained. It is, however, usually advisable to dilute the cooled solution to 200 c.c. with water free from carbon dioxide and, after filtering, to titrate 100 c.c.

The approximate neutralisation of the acid solution is very desirable, as in this way most of the sesquioxides are separated. There must, however, always be an excess of acid so as to avoid the precipitation of calcium hydroxide.

It is pointed out that calcium silicates, owing to the readiness with which they are decomposed, are practically basic compounds. Calcium, in the form of hydrated silicate (or even after being ignited for half an hour), can be determined by the above method. N. H. J. M.

Volhard's Copper Titration. OTTO KUHN (*Chem. Zeit.*, 1908, 32, 1056—1057).—The author agrees with Theodor (this vol., ii, 898) that Volhard's process gives very satisfactory results. He has, however,

introduced a slight modification so as to avoid the harmful influence of free nitric acid, which has a solvent action on the copper thiocyanate precipitate.

The solution of the alloy in nitric acid is mixed with ammonia until a permanent precipitate has formed, which is then redissolved by adding a decided excess of sulphurous acid; the solution is then heated to boiling, and precipitated with ammonium thiocyanate, the excess of which is estimated in the filtrate with silver solution as usual. The nitric acid may, of course, be expelled completely by evaporating with sulphuric acid, but this would render the course somewhat more complicated.

L. DE K.

Volumetric Estimation of Mercury by means of the Thiocyanate, Iodometric, and Acidimetric Processes. ERWIN RUPP (*Chem. Zeit.*, 1908, 32, 1077—1079).—*Thiocyanate Process.*—The solution, which must contain the mercury as mercuric nitrate or sulphate and be absolutely free from mercurous nitrate, also from chlorine and nitrous acid, is titrated with $N/10$ ammonium thiocyanate, using 2 c.c. of 10% iron-alum solution as indicator; 1 c.c. of thiocyanate = 0.01 gram of mercury. When the solution has been prepared by dissolving mercury in nitric acid, the oxidation is conveniently completed with potassium permanganate, the excess of which is then removed with a pinch of ferrous sulphate.

Iodometric Process.—Twenty-five to fifty c.c. of the solution, containing about 0.1—0.25 gram of the salt, are mixed in a stoppered flask with 1—2 grams of potassium iodide, 10—20 c.c. of 10% potassium hydroxide are added, and then 3 c.c. of 40% formaldehyde diluted with 10 c.c. of water. After shaking for two minutes, 10 c.c. of glacial acetic acid are added, and then 25 c.c. of $N/10$ iodine. When all traces of metallic mercury have disappeared, the excess of iodine is titrated with $N/10$ thiosulphate; no indicator is wanted. One c.c. of iodine solution = 0.01 gram of mercury.

Acidimetric Process.—This is based on the strong affinity of mercury for cyanogen. The mercury should be present as chloride, which may be effected, if necessary, by addition of 1 gram of potassium chloride; any free acid is carefully neutralised with potassium hydroxide, using phenolphthalein as indicator, and 20 c.c. of $N/2$ potassium cyanide are added. The excess of cyanide is then titrated with $N/2$ hydrochloric acid, using methyl-orange as indicator; 2 mols of cyanide = 1 at. of mercury. The titration may also be performed by simply adding the cyanide solution until the liquid turns pink.

Notwithstanding the great stability of the cyanide, it is completely decomposed by potassium iodide with formation of potassium cyanide, which may then be titrated with $N/2$ hydrochloric acid. Mercuric oxide may also be titrated with acid in presence of potassium iodide, from which it liberates the hydroxide. Instead of standardising the cyanide with $N/2$ acid, it may be also checked against a solution of mercuric chloride of known strength.

L. DE K.

Volumetric Estimation of Mercuric Oxide. ERWIN RUPP and W. F. SCHIRMER (*Pharm. Zeit.*, 1908, 53, 928).—Mercuric oxide

cannot be estimated by dissolving in hydrochloric acid and titrating the excess of this with alkali; but it may be titrated by dissolving about 0.25 gram in 10–20 c.c. of water containing 2–3 grams of potassium iodide, and titrating with *N*/10 hydrochloric acid, using methyl-orange as indicator. Heating on the water-bath promotes the solution of the oxide.

Another plan is to boil 0.25 gram of the oxide with 50 c.c. of water and 2 grams of mercuric cyanide. When all is dissolved, 1 gram of salt is added, and when cold the solution is titrated with *N*/10 hydrochloric acid, using methyl-orange as indicator.

The process may be employed for the testing of ointments. Two grams of the sample are boiled with 2 grams of mercuric cyanide and 50 c.c. of water until all the oxide has dissolved, 1 gram of salt is added, and the solution titrated with *N*/10 acid. On account of a slight saponification taking place, owing to the liberation of potassium hydroxide, the potassium iodide process is unsuitable for the testing of fatty ointments.

L. DE K.

Estimation of Manganese by means of Potassium Ferri-cyanide. HERMANN BOLLENBACH and E. LUCHMANN (*Chem. Zeit.*, 1908, 32, 1101–1102, 1114–1115).—The solution, which must be free from metals precipitable by hydrogen sulphide, and also from ferrous iron, cobalt, nickel, chromium, and reducing substances, is mixed with an excess of potassium ferricyanide. A decided excess of aqueous sodium hydroxide is added, and the manganese dioxide is collected and washed with hot water. The filtrate is acidified with excess of dilute sulphuric acid, and the potassium ferrocyanide formed in the reaction titrated with permanganate as usual. Two mols. of ferrocyanide = 1 at. of manganese.

In presence of ferrous iron, an aliquot part of the solution should be titrated with permanganate, and an allowance should be made.

L. DE K.

New Method of Attacking Ferro-compounds, particularly Ferro-silicon. PAUL NICOLARDOT (*Compt. rend.*, 1908, 147, 676–678).—In decomposing ferro-silicon by chlorine at a red heat, it is impossible to retain all the silicon chloride. The author finds that ferro-silicon is completely decomposed by heating with commercial sulphur chloride at 70° for three minutes. Ferro-titanium is somewhat less easily decomposed, whilst ferro-chromium requires a temperature above 120°. The process is carried out in a 250 c.c. flask, closed by a small graduated dropping funnel of special shape. The apparatus is evacuated, and exactly 2 c.c. of sulphur chloride cautiously introduced. On completion of the action which is started by heating, a few drops of ammonia solution are introduced, and the flask is filled up with water as it gradually cools. The products are estimated in the usual way.

R. J. C.

Separation of Tungstic Acid from Silica. PAUL NICOLARDOT (*Compt. rend.*, 1908, 147, 795–797).—This separation is most readily effected by heating the mixture at 500° in a current of air charged

with chloroform vapour, which, unlike carbon tetrachloride, does not give a deposit of carbon at this temperature. The tungsten is thus removed as a mixture of oxychlorides.

W. O. W.

Reduction of Stannic Oxide. DAVID B. DOTT (*Pharm. J.*, 1908, 81, 585).—When stannic oxide (0.15 gram) is heated with hypophosphorous acid (0.5 gram) over a Bunsen flame during thirty minutes, it is converted into stannous phosphate or pyrophosphate, which is readily soluble in warm hydrochloric acid. Silica remains unaffected by this treatment, and, if present, can be filtered from the hydrochloric acid solution of the tin.

T. A. H.

Physico-chemical Analysis of Mineral Water. ERNST HINTZ and LEO GRÜNHUT (*Zeitsch. angew. Chem.*, 1908, 21, 2359—2368. Compare *ibid.*, 1903, 16, 842).—A mathematical paper comprising a reply to Roloff's criticism of the formulæ used by the authors for calculating the middle dissociation value from the specific conductivity. The formulæ used by Roloff are deduced and shown to be only applicable in special cases, and a complete derivation of the authors' formula is given for the first time. Roloff's assumptions respecting the calculation of the freezing point are discussed, and examples given showing an error of +5.8% from the observed values when use is made of his mode of calculation.

J. V. E.

Assay of Turpentine and Estimation of Mineral Oil in Rosin Spirit. R. ADAN (*Bull. Soc. chim. Belg.*, 1908, 22, 389—396).—Herzfeld's sulphuric acid process is quite untrustworthy for the detection of petroleum products in turpentine or rosin spirit, but Burton's nitric acid method gives satisfactory results provided the temperature is lowered to -10° . Some samples, although pure, may still give 1—2% of insoluble oils.

Petroleum in turpentine or rosin spirit may also be detected by collecting the distillate passing over between 120° — 150° . In the absence of petroleum, the fraction is miscible in all proportions with aniline or acetic anhydride.

Pure turpentine should practically distil over at 162° . In the case of rosin spirit, the bulk of the distillate collects between 165° — 175° , and the distillation is not quite finished even at 180° . This fact facilitates its detection in mixtures. It may be also identified by Grimaldi's test with tin and hydrochloric acid, which gives a green coloration, and also by its odour.

L. DE K.

Estimation of Essential Oils in Spices. R. REICH (*Zeitsch. Nahr. Genussm.*, 1908, 13, 497—509).—The method described by Mann (*Abstr.*, 1902, ii, 432) gives trustworthy results if the point at which the solvent has been removed completely can be exactly determined, and for this purpose the author recommends the following modification as giving the best result. The solution of the ethereal oil in ether, or pentane, is placed in the evaporation flask, and the solvent is evaporated almost completely. A few drops of isopropyl

chloride are then added to the flask, and the drying is continued until the current of air and other gases issuing from the platinum jet no longer gives a green flame when allowed to impinge against a heated copper gauze. The method is trustworthy for estimations of essential oils of cinnamon, cassia, cloves, peppermint, aniseed, thyme, ginger, and camphor, but cannot be used in the case of oils of carraway, lemon, eucalyptus, and turpentine, as these contain extremely volatile substances.

W. P. S.

Detection of Small Quantities of Methyl Alcohol in the Presence of Ethyl Alcohol. LEONHARDT E. HINKEL (*Analyst*, 1908, 33, 417—419).—The following method, in which the alcohols are oxidised to their corresponding aldehydes, and the formaldehyde then detected by means of morphine hydrochloride, is stated to be capable of detecting the presence of methyl alcohol in ethyl alcohol when the proportion of the former alcohol is not less than 5%. One c.c. of the mixed alcohols is placed in a small distilling flask, and the oxidising agent is added. If ammonium persulphate is used, 0.8 gram of the salt is added, followed by 3 c.c. of dilute sulphuric acid (1:5); or in the case of potassium dichromate, 1.5 grams of the salt and 1.5 grams of pure sulphuric acid are employed. In both cases, the mixture is diluted with water to 20 c.c. and distilled, the distillate being collected in test-tubes in five separate portions of 2 c.c. each. The first two portions, which will contain all the acetaldehyde, are rejected; to each of the remaining portions are added a few drops of 0.5% morphine hydrochloride solution, and concentrated sulphuric acid is run into each tube so as to form a layer at the bottom. In the presence of formaldehyde (resulting from the oxidation of the methyl alcohol), a violet ring will be formed at the junction of the two liquids. Pure ethyl alcohol always yields a trace of formaldehyde on oxidation, but the reaction obtained when 5% of methyl alcohol is present cannot be confused with the coloration due to the ethyl alcohol.

W. P. S.

Apparatus for Polarising at 87°. ALBERT P. SY (*J. Amer. Chem. Soc.*, 1908, 30, 1790—1791).—Apparatus is described for determining the rotatory power of sugar solutions at 87°. It consists essentially of a jacketed polariscope tube heated by means of a current of water from an instantaneous water heater. For details, the description and diagram in the original must be consulted.

E. G.

Influence of Clarification with Lead Acetate on the Estimation of Invert Sugar. O. SCHREFELD (*Zeitsch. Ver. deut. Zuckerind.*, 1908, 634, 947—956).—It has been shown by Prinseer-Geerligs (this vol., ii, 991) and others that, under certain conditions, laevulose and, in less degree, dextrose are partly precipitated from their aqueous solutions by basic lead acetate. The author has carried out experiments to ascertain whether the use of this reagent for clarifying solutions of commercial sugar products interferes with the estimation of invert sugar by the reduction of Fehling's solution.

The results show that the reducing power of invert sugar may be lowered by basic lead acetate, this being especially the case with low products and with high contents of invert sugar. When neutral lead acetate is employed, there is, however, little danger of low results being obtained.

T. H. P.

Optically Active Non-saccharine Substances in Sugar Beet which are Eliminated by the Action of Lime in the Purification of the Sap, and their Polarimetric Estimation. FRANZ HERLES (*Zeitsch. Zuckerind. Böhm.*, 1908, 33, 94—98).—Polarimetric determinations in beet juice before and after boiling with lime generally resulted in lower figures after treatment with lime. The differences varied between 0.0% and 0.4%. Beet juice therefore contains, as a rule, some optically active non-sugar which is either precipitated or destroyed by the lime employed during the process of purification.

N. H. J. M.

Colour Reactions of the Carbohydrates Based on the Formation of Furfuraldehyde from them. Reactions with Indole and Carbazole. C. FLEIG (*J. Pharm. Chim.*, 1908, [vi], 28, 385—392).—When 0.5 c.c. of a dilute solution of sucrose or other carbohydrate (many proteins also react) is treated with 3—4 c.c. of pure hydrochloric acid, the mixture boiled momentarily (if any coloration is thereby produced the carbohydrate solution should be diluted and less of it used), and 3—4 drops of a 0.1% alcoholic solution of indole added, a yellow-orange or reddish-orange coloration is produced. One to two drops of a 0.01% solution of sucrose diluted to 0.5 c.c. will give this reaction. Most of the sugars, starches, dextrins, glucosides, etc., react, but the polybasic alcohols, sorbitol, dulcitol, etc., are inactive. If sulphuric acid is used in place of hydrochloric acid, a blank experiment containing no carbohydrate must be made for comparison. The reaction with carbazole (which is given by the same substances as give the indole reaction) is obtained by adding 1—2 drops of a saturated alcoholic solution of carbazole and 1 c.c. of pure sulphuric acid to 0.5 c.c. of the carbohydrate solution, when a reddish-violet ring is formed at the junction of the two liquids. A blank experiment is necessary also in this case, since at certain temperatures carbazole and sulphuric acid react, giving red or violet colorations.

E. H.

Polarimetric Estimation of Starch. CARL J. LINTNER (*Zeitsch. Nahr. Genussm.*, 1908, 16, 509—512).—Sulphuric acid may be used in place of hydrochloric acid in the method described previously by the author (*Abstr.*, 1907, ii, 823). 2.5 Grams of the finely-ground flour are mixed in a mortar with 10 c.c. of water and 20 c.c. of sulphuric acid, D 1.7 (77%); at the end of twenty-five minutes the mass is washed into a 100 c.c. flask by the aid of dilute sulphuric acid (1 : 3), 5 c.c. of 8% phosphotungstic acid solution are added, and the process then continued as described (*loc. cit.*). When sulphuric acid is used, barley starch has $[\alpha]_D^{20}$ 191.7°, and this value differs for each kind of starch. Unless the value be determined for each starch, the hydrochloric acid method is to be preferred, as, in this case, the value is fairly constant.

W. P. S.

Estimation of Tartaric Acid in Wines by Evaporation. W. MESTREZAT (*Ann. Chim. anal.*, 1908, 13, 433—436).—The evaporation method proposed by Pasteur and modified by Reboul is considered to be more trustworthy than the official (French) method for the estimation of tartaric acid in wines. The following way of carrying out the estimation is recommended: 50 c.c. of the wine are evaporated to such an extent that the residue, when cold, is semi-fluid. After the lapse of five days, the crystals of potassium hydrogen tartrate which have formed are washed with 40% alcohol saturated previously with potassium hydrogen tartrate, and are then titrated in the usual manner.

W. P. S.

Estimation of Malic Acid. M. EMMANUEL POZZI-ESCOFFIER (*Bull. Assoc. chim. Sucr. Dist.*, 1908, 26, 266—267*).—It is pointed out by the author that the untrustworthiness of both the American official method and the process described by Cowles (*Abstr.*, 1908, ii, 904) for the estimation of malic acid is mainly due to the use of precipitants which are insoluble in alcohol, and that, at the same time, calcium malate is appreciably soluble in 85% alcohol. Malic acid is best precipitated by means of a solution of barium bromide in 96% alcohol, the solution being rendered slightly ammoniacal before use, and the precipitation made in the presence of an excess of alcohol.

W. P. S.

Detection of Benzoic Acid in Butter. LUCIEN ROBIN (*Ann. Chim. anal.*, 1908, 13, 431—433).—The author modifies the method described by Halphen (*Abstr.*, 1908, ii, 906) in order to prevent the formation of an emulsion when the butter is extracted. A portion of the butter is melted together with 50 c.c. of water, 15 c.c. of alcohol, and 0.5 gram of sodium hydrogen carbonate; the aqueous portion is then separated, acidified with sulphuric acid, heated, and filtered. The filtrate is shaken with ether, and the ethereal extract, after being washed with a mixture of 20 c.c. of water and 5 c.c. of alcohol, is shaken with 25 c.c. of the same water-alcohol mixture to which has been added 0.3 gram of sodium hydrogen carbonate. The aqueous portion, containing the benzoic acid as its sodium salt, is separated and evaporated to dryness; the residue is heated with 5 c.c. of sulphuric acid and 10 drops of fuming nitric acid until sulphuric acid fumes are given off, and the solution is then poured into 50 c.c. of cold water. After rendering the solution ammoniacal, a few drops of ammonium sulphide are added, when an orange-red coloration develops rapidly if the butter contains benzoic acid.

W. P. S.

Colour Reactions of Aromatic Aldehydes with Phenols and Various Cyclic, Heterocyclic, and Open-chain Compounds. C. FLEIG (*Bull. Soc. chim.*, 1908, [iv], 3, 1038—1045).—When an acid is added to a solution of an aromatic aldehyde in alcohol also containing one of a variety of substances of which the following may be mentioned as types, phenol, gallic acid, camphor, menthol, aniline, pyrrole, indole, mercaptan, and isobutyl alcohol, a coloration, usually yellow, orange, red, or violet, or in some cases a

* and *Bull. Soc. chim. Belg.*, 1908, 22, 413—414.

play of colours, is produced. The acid used may be sulphuric, hydrochloric, lactic, or a mixture of the last two. The colours given by menthol and terpin are similar to those yielded by cholesterol and biliary acids.

The aminophenols yield orange-red colorations, which turn yellow and finally disappear on addition of excess of acid. With alkalis they also change to yellow, but on the further addition of acid in excess become red. These changes are explained by assuming the existence in the solution of a tautomeric substance *d*; this, on addition of sodium hydroxide, forms a product *Nad*, which is yellow in presence of excess of alkali and red in presence of excess of acid.

These colour reactions can be applied in the detection of free hydrochloric acid in gastric juice, and possibly to the detection of mineral acids in adulterated wines.

T. A. H.

Colorimetric Estimation of Benzaldehyde in Almond Extracts. ALPHEUS G. WOODMAN and E. F. LYFORD (*J. Amer. Chem. Soc.*, 1908, 30, 1607—1611).—The reagents required are magenta decolorised by sulphurous acid and alcohol free from aldehyde. The former is prepared by dissolving 0.5 gram of magenta in 100 c.c. of water, and adding a solution containing 20 grams of sulphur dioxide. When decolorised, the solution is diluted to one litre. The alcohol is purified by distilling over silver oxide. To the distillate are added 15 grams of *m*-phenylenediamine hydrochloride per litre, a rapid current of air is drawn through the solution for three hours, and the alcohol is again distilled, the first 100 c.c. being rejected. The method as used with commercial almond extract may be outlined as follows:

Ten grams of the sample are diluted to 50 c.c. with the purified alcohol. Of this, 2 c.c. are placed in a Mohr cylinder and diluted with alcohol to 20 c.c. Three standard solutions are made up by diluting 2, 4, and 6 c.c. of benzaldehyde solution (alcohol containing 1 mg. of benzaldehyde per c.c.) to 20 c.c. and placing them in similar colorimeter tubes. The tubes are then placed for some time in water at 15°, and to the contents of each are added rapidly 10 c.c. of the magenta reagent, also at 15°. After ten minutes, the unknown sample is matched with the nearest standard in the usual manner by withdrawing part of either liquid. The depth of colour is proportional to the amount of benzaldehyde present.

L. DE K.

Detection of "Saccharin" (*o*-Benzoic-sulphinide) and other Artificial Sweetening Materials in Beverages and Foods. ALBERTO BIANCHI and ETTORE DI NOLA (*Boll. chim. farm.*, 1908, 47, 599—605). Compare this vol., ii, 440).—The authors give the following modification of the method devised by Villiers and others (compare Abstr., 1904, ii, 599) for the detection of *o*-benzoic-sulphinide in foods. The liquid, or, in the case of a solid, a suitable liquid extract, is freed from alcohol, heated to boiling, and acidified with about 20 drops of acetic acid per 100 c.c. The liquid is shaken, cooled, and mixed with about 10 c.c. of 20% lead acetate solution per 100 c.c. After half an hour, the excess of lead is precipitated by means of a solution containing 10% of sodium sulphate and 10% of

sodium phosphate, double the volume of lead acetate used being always sufficient. The filtered liquid, concentrated to 70–80 c.c. if necessary, is acidified with 6–8 c.c. of dilute sulphuric acid (1:3), and shaken in a separating funnel with its own volume of a mixture of equal parts of ether and benzene. The benzene-etheral extract is then slowly evaporated in a flat-bottomed glass dish. The residue is tested for (1) *o*-benzoisulphinide by tasting, and by fusion with sodium hydroxide at 270°; (2) salicylic acid by extracting with a small quantity of alcohol, diluting the alcoholic solution, and adding ferric chloride. If salicylic acid is found, it must be destroyed before the residue is tested for *o*-benzoisulphinide (compare Villiers, etc., *Abstr.*, 1904, ii, 599).

The above method also serves for the detection of the ammonium ("sucramine"), sodium ("sucrose"), and magnesium derivatives of *o*-benzoisulphinide, and of the so-called extract of sugar-cane, which is a solution of *o*-benzoisulphinide in glycerol.

"Dulcine" or "sucrol" (*p*-phenetolcarbamide) may be detected by treating a small portion of the residue left by the benzene-etheral extract (above) with silver nitrate (compare Ruggeri, *Ann. Lab. Centr. Gabelle*, 3, 143).

T. H. P.

Microchemical Studies. A. BOLLAND (*Monatsh.*, 1908, 20, 965–994).—A paper dealing with the microchemical detection of the following substances: methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, triethylamine, propylamine, amylamine, hexylamine, neurine, ethylenediamine, pentamethylenediamine, choline, betaine, methylguanidine, α -aminovaleric acid, parvoline, pilocarpine, piperine, coniine, conhydrine, ψ -conhydrine, hyoscyne, cornutine, ergotinine, colchicine, emetine, lobelline, solanine, solanidine, chelidonium, chelerythrine, sanguinarine, strophanthine, digitalin, picrotoxin, and santonin. The appearance, crystallographic and optical properties of the precipitates obtained on treating the tartrates of these substances with various reagents are described.

The refractive indices of the following alkaloids, determined by the immersion method, are given: solanine, solanidine, colchicine, conhydrine, ψ -conhydrine, and ergotinine; also of the tartrates of morphine, thebaine, quinine, cinchonidine, coniine, nicotine, hydrastine and cocaine.

W. H. G.

Colour Reactions of Proteins. C. FLEIG (*Ann. Chim. anal.*, 1908, 13, 427–431).—The following colour reactions are given by the proteins which contain a carbohydrate group in their molecule, namely, ovalbumin, ovoglobulin, serum-albumin, and particularly the glucoproteins (mucin). The test is best carried out by mixing a few drops of a 20% solution of the reagent with a dilute solution of the protein and pouring the mixture on to the surface of a little concentrated sulphuric acid contained in a test-tube. Orcinol, catechol, pyrogallol, phenol, menthol, camphor, terpene, carbazole, thiophen, and pyrrole give red colorations; resorcinol and indole blue, and phloroglucinol, reddish-brown.

W. P. S.

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- of Finggi, near Anticoli, physico-chemical investigation of the (NASINI and LEVI), A., ii, 401.
- of Kissingen, radioactivity of the (JENTZSCH), A., ii, 9, 143.
- of Lavez-les-Bains, radioactivity of the (SARASIN, GUYE, and MICHELI), A., ii, 143.
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ERRATA.

COLLECTIVE INDEX, 1873—1882 (AUTHORS).

al i for "773" read "173."

VOL. LXX (ABSTR., 1896).

PART I.

for " $\text{COOEt}\cdot\text{C}(\text{OH})(\text{CO}\cdot\text{NHPh})\cdot\text{C}(\text{OH})(\text{CO}\cdot\text{NHPh})\cdot\text{COOEt}$ "
read " $\text{COOEt}\cdot\text{CH}(\text{O}\cdot\text{CO}\cdot\text{NHPh})\cdot\text{CH}(\text{O}\cdot\text{CO}\cdot\text{NHPh})\cdot\text{COOEt}$."

VOL. LXXIV (ABSTR., 1898).

PART I.

for "Methoxytolualdehyde" read "Ethoxytolualdehyde."
delete "whilst the isomeride."
for ".OEt" read ".OMe."
delete "third."
for "OEt" read "OMe."

COLLECTIVE INDEX, 1893—1902.

PART II (Subjects).

vol. ii insert "Ethoxytolualdehyde (GATTERMANN AND FRENZEL),
1898, I, 477."
,, ii for "phenyltoluidine" read "phenyltolylamine."
,, i delete "See also Methyl diphenylamine."

VOL. LXXXVIII (ABSTR., 1905).

PART I.

for "arylsulphonates" read "arylsulphinates."

PART II

7*, 9*, 11*, 19* for "rice" read "rye."

VOL. XC (ABSTR., 1906).

PART I.

for "Georg" read "Wilhelm."

PART II.

for "the sulphide" read "iron."
al. i }
, if for "Georg" read "Wilhelm."

* From bottom.

ERRATA (continued).

VOL. XCII (ABSTR., 1907).

PART I.

Page	Line	
137	1	for "2-Nitroisophthalic acid" read "2-Nitroisophthalic acid."
233	16	„ "phenylrhodanic" read "anilinorhodanic."
509	9	„ "hexahydro- <i>m</i> -toluic acid" read "1-methylcyclohexane-2-carboxic"

PART II (INDEX).

1009	19*	col. ii	for "602" read "622."
1059	10	„ i	„ "Köfler" read "Löfler."
1068	10*	„ i	insert "an attempt to synthesise collidine, A., i, 14)
1074	2	„ ii	for "Mayer" read "Meyer."
1076	10	„ ii	insert "reversible reactions of the first order, A., ii."

VOL. XCIV (ABSTR., 1908).

PART I.

44	5*	for " $C_{12}H_{20}N_2MeI_3HI.H_2O$ " read " $C_{12}H_{20}N_2MeI.HI.H_2O$."
219	5	„ " $CHMe_2CO.CMe:CH_2$ " read " $CHMe_2CO.CMe:CH_2$."
266	19*	„ "diethylaminochyl" read "diethylamino-propyl."
275	8*	„ "2-Phenylpyrrolidine" read "2-Phenylpyrrolidine."
288	13, 14	„ "3-phenyl-1-methyl-2-quinoline" read "3-phenyl-1-methyl-2-quinoline."
315	16*	„ "GIALDINO" read "GIALDINI."
406	11*	„ "1-isopropylhexano-2-one" read "1-isopropylcyclohexano-2-one."
408	21	„ "ROELKER" read "RÖLKER."
457	20*	„ "4-nitro-4-hydroxydiphenylamine" read "4-nitro-4'-hydroxydiphenylamine."
474	4	„ "aminophenylthiocarbamide" read "aminophenylcarbamide."
494	10*	„ " HCl_2Cl " read " $CHCl_2$."
590	6*	„ " $NMe_2.C_6H_4.As(OH).ONa$ " read " $NMe_2.C_6H_4.AsO_3$."
591	20, 21	„ "crystallising" read "the sodium salt crystallises."
591	5*	„ "KUNX" read "KAHN."
652	21	„ " $CH_2.CO \begin{matrix} \nearrow NH.C_6H_4(CO_2Me)_2 \\ \searrow CH_2.CO \end{matrix}$ " read " $CH_2.CO \begin{matrix} \nearrow CH_2.CO \\ \searrow CH_2.CO \end{matrix} N.C_6H_4$."
687	4	„ "1899" read "1889."
692	21*	„ "VIII" read "XXIX."
765	17	delete "FUSIA."
773	7*	for " α -Bromoisoheptyl- β -aminobutyric acid" read " α -Bromoisoheptyl- β -amino- γ -hydroxybutyric acid."
809	1*	} „ " <i>dl</i> -fenchene" read " <i>D</i> -l-fenchene."
810	13	
810	17	
810	23	
811	1	
810	4	
810	5	} „ " <i>dl</i> -hydroxyfenchene acid" read " <i>D</i> -l-hydroxyfenchene."
810	9	
811	6	
810	12*	
927	1, 2, 3	should read "p-Nitrobenzenesulphonic acid, m. p. 256°; it is proved by its yielding aminosulphonic acid at enediamine when reduced by sodium hyposulphite aqueous solution."
927	13	for "hydrogen" read "hyposulphite."

* From bottom.

ERRATA (continued)

For "diaminodiphenyl derivative of carbamide" read
"diaminodiphenylcarbamide derivative."
For "p-aminophenol" insert "diazotised."
For "aminosalicylic acid" insert "diazotised."
For "X" read "XXXIV."
For "not" read "now."

PART II.

for "GUILLAUME SCHAEFFER" read "GEORGES SCHAEFFER."
 "BAYLIS" read "BAYLISS."
 "190" read "1907."
 "TITRATION OF FORMALDEHYDE" read "Formaldehyde-titration."
 15. "Formaldehyde may be titrated with alkali and phenolphthalein as indicator" read "Sørensen's so-called 'formaldehyde-titration' (this vol., i, 115) may be carried out."
 "usual titration" read "formaldehyde-titration."
 "C. S." read "G. S."
 "J. Landw." read "J. exper. Landw."
 "one inch" read "nine inches."
 "517" read "644."
 6* The statement and criticism are wrongly attributed. The statement is by Knoop, the criticism by Friedmann.
 for "1908" read "1898."
 "ALLALET" read "ALLARD."

* From bottom.

INSTRUCTIONS TO ABSTRACTORS,

GIVING THE

ENCLOSURE AND SYSTEM OF NOTATION

ADOPTED IN THE ABSTRACTS.

One of the objects of the abstracts of chemical papers published elsewhere in the Transactions of the Society is to furnish the Fellows with an account of the progress of chemical science from month to month. It must be understood that as the abstracts are prepared for the information of the Fellows in general, they cannot possibly be so full or so detailed as to obviate on the part of those who are engaged in special investigations the necessity of consulting the original memoirs.

The abstracts of papers must be given literally.

When beginning to write the abstract, the whole of the original paper should be read, in order that a judgment may be formed of its scope and of the scale on which the abstract should be made. In the case of papers dealing with subjects not strictly chemical, the abstract should refer only to matters of chemical interest in the

The abstract should consist mainly of the expression, in the author's own words, of the substance of the paper.

The abstract should be made as short as is consistent with a concise and accurate statement of the author's results.

A concise statement showing the general trend of the investigation should be given at the commencement of those abstracts where the original permits of it.

In the case of an abstract of a paper on the same subject, either by the author of the paper abstracted, or by some other author, has already been abstracted, note should, as a rule, be made of this fact.

A paper which has appeared once in the *Abstracts* is not to be reabstracted; again, a reference being given to the volume in which the paper may be found.

As a rule, details of methods of preparation or analysis, or of the mode of speaking of work, are to be omitted, unless such details are necessary to the understanding of the results, or have some independent value. Further, comparatively unimportant compounds, such as organic salts of organic bases or acids, should be mentioned only. On the other hand, data such as melting and boiling points, specific rotation, &c., must be given in every case unless they have been given in earlier papers.

Nomenclature.

10. Employ names such as *sodium chloride*, *potassium nitrate*, etc., for inorganic compounds, and use the terminals *ous* and *ic* in distinguishing compounds of different orders derived from the same elementary radicle; such, for instance, as mercurous and mercuric chlorides, sulphurous and sulphuric acids.

11. Term compounds of metallic radicles with the terminal *hydroxides* and not *hydrates*, the name *hydrate* being reserved for compounds supposed to contain water of combination or crystallization.

12. Term salts containing an amount of metal equivalent to the replaceable hydrogen of the acid, *normal* and not *neutral*; assign names such as sodium hydrogen sulphate, disodium hydrogen phosphate, &c., to the acid salts. Basic salts as a rule are designated merely by their *formule*.

13. Names in common use for oxides should be employed. Example: NO, nitric oxide; CO₂, carbon dioxide; P₄O₁₀, phosphoric oxide; As₂O₃, arsenious oxide; Fe₂O₃, ferric oxide.

14. In open chain compounds, Greek letters must be used to indicate the position of a substituent, the letter α being assigned to the carbon atom in the formula, except in the case of CN or other groups. For example, CH₃·CH₂·CH₂·CH₂·I α -iodobutane, CH₃·CH₂·C≡CH α -cyanopropene.

15. Isomeric open chain compounds are most conveniently represented as substitution derivatives of the longest carbon chain. For example,

$\begin{array}{c} \text{CH}_3 \\ | \\ \text{CH}_3\text{CH}_2\text{CH} > \text{CH} \cdot \text{CH} < \begin{array}{c} \text{CH}_3 \\ | \\ \text{CH}_3 \end{array} \end{array}$ or CH₃·CH₂·CHMe·CHMe should be termed β -dimethylpentane, not methyl-ethyl-methane, and $\begin{array}{c} \text{CH}_3 \\ | \\ \text{CH}_3\text{CH} > \text{CH} \cdot \text{CH} < \begin{array}{c} \text{CH}_3 \\ | \\ \text{CO}_2\text{H} \end{array} \end{array}$ or CH₃·CHMe·CH(CO₂H)₂ should be termed $\alpha\beta$ -dimethylbutyric acid, not $\alpha\beta\beta$ -trimethylbutyric or α -methylisovaleric, or methylisopropylacetic acid.

16. Use names such as methane, ethane, &c., for the paraffin or hydrocarbon series of the C_nH_{2n+2} series of CH₄, (CH₂)_n, &c. Term the hydrocarbons C₂H₂ and C₃H₄ ethene and acetylene respectively (not ethene and ethine). For the ethylene series are to be indicated by the suffix *-ene*, and for the acetylene series, wherever possible, by *-yne*. Adopt the name *allene* for the hydrocarbon CH₂·C=CH₂.

17. Distinguish all hydroxyl derivatives of hydrocarbons ending in *ol*. Alcohols should be spoken of as monohydric, according to the number of OH-groups. Compounds are not alcohols, but for which names ending in *ol* have been assigned, are to be represented by names ending in *ol*, if a system cannot be given, thus anisole not anisol, indole not indol. Such as MeONa, EtONa, &c., should be termed sodium methyl ethoxide, &c.

18. The *radicles* indicated in the name of a compound

the order fluoro-, chloro-, bromo-, iodo-, nitro-, nitroso-, amino-, cyano-, thiocyno-, hydroxy-, keto-.

Compounds analogous to the acids of the lactic series containing hydroxy-groups should be termed *hydroxy-*derivatives, and not *oxy-*derivatives; for example, hydroxyacetic and not oxyacetic acid. Compounds containing the analogous groups OEt, OPh, OAc, &c., should in like manner be termed ethoxy-, phenoxy-, acetoxy-derivatives. Thus ethyl-lactic acid, $\text{OEt}\cdot\text{CHMe}\cdot\text{CO}_2\text{H}$, instead of ethyl-lactic acid; diethylproto-*l*-benzoic acid, $(\text{OEt})_2\text{C}_6\text{H}_3\cdot\text{CO}_2\text{H}$, instead of diethylproto-*l*-benzoic acid; and *o*-acetoxypropionic acid, $\text{OAc}\cdot\text{CHMe}\cdot\text{CO}_2\text{H}$, instead of *o*-acetoxypropionic acid. Terms such as diethylprotocatechuic acid should be understood to mean a compound formed by the displacement of hydroxyl groups in the hydrocarbon radicle of protocatechuic acid by ethyl groups, $\text{C}_6\text{H}_3\text{Et}_2(\text{OH})_2\cdot\text{CO}_2\text{H}$, and not $\text{C}_6\text{H}_3(\text{OEt})_2\cdot\text{CO}_2\text{H}$, just as protocatechuic acid is understood to be the name of a compound with the formula $\text{C}_6\text{H}_3\text{Br}_2(\text{OH})_2\cdot\text{CO}_2\text{H}$.

The term *ether* should be restricted to the oxides of hydrocarbons and their derivatives, and the esters (so-called compounds or ethereal salts) should be represented by names similar to those given to metallic salts.

When a substituent is one of the groups NH_2 , NHR , NR_2 , NH or N , the name should end in *ine*; for example, β -aminopropionic acid, $\text{H}_2\text{N}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, β -anilino-acrylic acid, $\text{NHPh}\cdot\text{CH}=\text{CH}\cdot\text{CO}_2\text{H}$, *o*-anilino-*l*-propionic acid, $\text{NH}\cdot\text{CMe}\cdot\text{CO}_2\text{H}$.

Compounds of the radicle SO_3H should, whenever possible, be termed sulphonic acids, or failing this, sulpho-compounds; for example, *o*-sulphonic acid, sulphobenzoic acid.

Basic substances should invariably be indicated by names ending in *ine*, as aniline instead of anilin, the termination *in* being used for certain neutral compounds, viz., glycerides, glucosides, alkaloids, and proteins, such as palmitin, amygdalin, albumin. Compounds of basic substances with hydrogen chloride, bromide, iodide, &c., should always receive names ending in *ide* and not *ate*, as hydrochloride and not morphine hydrochlorate.

The Collective Index, 3rd decade (1893-1902) should be adopted as the standard of reference on questions of nomenclature not provided for in the preceding sections.

Notation.

In empirical formulae the elements are to be given in the order O, N, Cl, Br, I, F, S, P, and the remainder alphabetically. Abbreviations should be omitted unless essential to the understanding of the results; as a rule, they should not be written on a separate line, but should "run on" with the text.

To economise space, it is desirable:

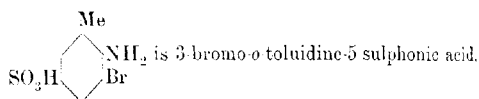
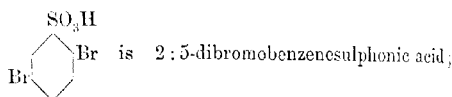
- 1) That *dots* should be used instead of *dashes* in connecting contiguous symbols or radicles, whenever this does not interfere with the clearness of the formula.

- (b) That formulæ should be shortened by the employment of the symbols Me for CH_3 , C_2H_5 , Pr^a for $\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_3$, Pr^s for $\text{CH}_3(\text{CH}_2)_2$ for C_6H_5 , Py for $\text{C}_5\text{H}_4\text{N}$, Ac for $\text{CO}\cdot(\text{CH}_3)_2$, and CO-C₆H₅.

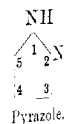
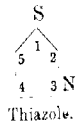
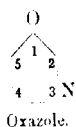
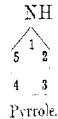
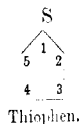
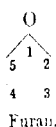
- (c) That formulæ should be written *in one line* so that this can be done without obscuring their meaning.

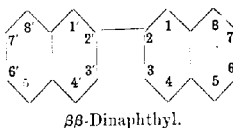
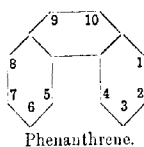
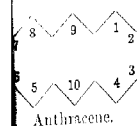
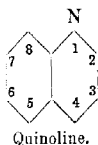
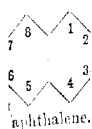
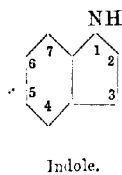
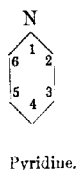
28. In representing the constitution of benzene derivatives the relative positions of the radicles in the symbol of benzene should be indicated by numerals, instead of by means of the hexagonal figure.

- (a) The abbreviations *o*-, *m*-, and *p*-, should be used; of 1:2- or ortho-, 1:3- or meta-, and 1:4- or *p*-.
 (b) In numbering positions in the case of substitution derivatives of phenol, aniline, benzonitrile, benzenesulphonic acid, benzaldehyde, and the characteristic radicle of each of these substances is to be regarded as in position 1 (Collective Index).
 (c) Names of substitution derivatives should be such a way that the position of the substituent is indicated by a numeral prefixed; for example



29. In representing the constitution of derivatives of other "chain" hydrocarbons, graphic formulae should not be employed; the system of numbering positions indicated in Richter's *Lehrbuch der Kohlenstoff-Verbindungen* (2nd edition 1899, pp. 16-26) should be used, of which the following schemes may be regarded as typical.





Manuscript.

view of the difficulty of dealing with MSS. of widely varying
abstracts cannot be accepted unless written on quarto paper

more than one abstract must appear on a sheet.

When an abstract exceeds a sheet in length, the sheets must be
together by means of a pin at the top left-hand corner.

The name of the abstractor must be written diagonally at the
bottom corner of the first sheet of the abstract.

Proofs.

Abstractors are expected to read and correct proofs carefully,
checking all formulae and figures against MSS.

Proofs, however small, must be returned to the Sub-Editor
within 24 hours after receipt from the printers.

The Editor's decision, in all matters connected with the
manuscript, must be considered final.

The numbering, proposed originally by E. Fischer, is adopted in the text of the

JOURNALS FROM WHICH ABSTRACTS ARE MADE

All references to Journals should give the abbreviated title, the year of publication, the series, the volume and the page; thus *Ber.* 1901, **34**, 2455; *Bull. Soc.* 1901, [iii], **25**, 794; *Gazzetta* 1901, **31**, i, 554.

ABBREVIATED TITLE.	JOURNAL.
<i>Amer. Chem. J.</i> . . .	American Chemical Journal.
<i>Amer. J. Pharm.</i> . . .	American Journal of Pharmacy.
<i>Amer. J. Physiol.</i> . . .	American Journal of Physiology.
<i>Amer. J. Sci.</i> . . .	American Journal of Science.
<i>Anal. Fis. Quim.</i> . . .	Anales de la Sociedad Española Física y Química.
<i>Analyst</i> . . .	The Analyst.
<i>Annalen</i> . . .	Justus Liebig's Annalen der Chemie.
<i>Ann. Physik</i> . . .	Annalen der Physik.
<i>Ann. Chim. anal.</i> . . .	Annales de Chimie analytique appliquée à l'Analyse à l'Agriculture, à la Pharmacie et à la Médecine.
<i>Ann. Chim. Phys.</i> . . .	Annales de Chimie et de Physique.
<i>Ann. Inst. Pasteur</i> . . .	Annales de l'Institut Pasteur.
<i>Ann. sci. Univ. Jassy</i> . . .	Annales scientifiques de l'Université de Jassy.
<i>Arch. exp. Path. Pharm.</i> . . .	Archiv für experimentelle Pathologie und Pharmacologie.
<i>Arch. Hygiene</i> . . .	Archiv für Hygiene.
<i>Arch. Néerland.</i> . . .	Archives Néerlandaises des sciences exactes et naturelles.
<i>Arch. Pharm.</i> . . .	Archiv der Pharmazie.
<i>Arch. Sci. phys. nat.</i> . . .	Archives des Sciences physiques et naturelles.
<i>Arkiv Kem. Min. Geol.</i> . . .	Arkiv för Kemi, Mineralogi och Geologi.
<i>Atti R. Accad. Sci. Torino.</i> . . .	Atti della Reale Accademia delle Scienze di Torino.
<i>Atti R. Accad. Lincei</i> . . .	Atti della Reale Accademia dei Lincei.
<i>Beitr. chem. Physiol. Path.</i> . . .	Beiträge für chemische Physiologie und Pathologie.
<i>Ber.</i> . . .	Berichte der Deutschen chemischen Gesellschaft.
<i>Ber. Deut. bot. Ges.</i> . . .	Berichte der Deutschen botanischen Gesellschaft.
<i>Ber. Deut. pharm. Ges.</i> . . .	Berichte der Deutschen pharmazeutischen Gesellschaft.
<i>Ber. Deut. physikal. Ges.</i> . . .	Berichte der Deutschen physikalischen Gesellschaft.
* <i>Bied. Zentr.</i> . . .	Biedermann's Zentralblatt für Agrikulturchemie und rationellen Landwirtschafts-Betrieb.
<i>Bio-Chem. J.</i> . . .	The Bio-Chemical Journal.
<i>Biochem. Zeitsch.</i> . . .	Biochemische Zeitschrift.
<i>Bull. chim. farm.</i> . . .	Bollettino chimico farmaceutico.
<i>Bull. Acad. roy. Belg.</i> . . .	Académie royale de Belgique—Bulletin des Sciences.
<i>Bull. Acad. Sci. Cracow</i> . . .	Bulletin international de l'Académie des Sciences de Cracovie.
<i>Bull. Acad. Sci., St. Pétersbourg.</i> . . .	Bulletin de l'Académie Impériale des Sciences de Pétersbourg.
<i>Bull. Assoc. chim. Sver. Dist.</i> . . .	Bulletin de l'Association des chimistes de Suède de Distillerie.
<i>Bull. Coll. Agr. Tokyo</i> . . .	Bulletin of the College of Agriculture, Imperial University, Tokyo.
<i>Bull. Geol. Soc. Amer.</i> . . .	Bulletin of the Geological Society of America.
<i>Bull. Imp. Inst.</i> . . .	Bulletin of the Imperial Institute.
<i>Bull. Soc. chim.</i> . . .	Bulletin de la Société chimique de France.
<i>Bull. Soc. chim. Belg.</i> . . .	Bulletin de la Société chimique de Belgique.
<i>Bull. Soc. chim. Min.</i> . . .	Bulletin de la Société française de Minéralogie.
<i>Bull. Soc. ind. Mulhouse</i> . . .	Bulletin de la Société industrielle de Mulhouse.
<i>Centr. Bakt. Par.</i> . . .	Centralblatt für Bakteriologie, Parasitenkunde und Infektionskrankheiten.
<i>Centr. Min.</i> . . .	Centralblatt für Mineralogie, Geologie und Petrologie.
* <i>Chem. Zentr.</i> . . .	Chemisches Zentralblatt.

* Abstracts from the *Zentralblatt* are made only in the case of papers published in journals other than those included in this list.

JOURNALS FROM WHICH ABSTRACTS ARE MADE.

ABSTRACTED TITLE.	JOURNAL.
Chem. News.	Chemical News.
Fett- u. Harz-Ind.	Chemische Revue über die Fett- und Harz-Industrie.
Chim. Weekblad.	Chemisch Weekblad.
Chemiker Zeitung.	Chemiker Zeitung.
Comptes rendus hebdomadaires des Séances de l'Académie des Sciences.	Comptes rendus hebdomadaires des Séances de l'Académie des Sciences.
Experiment Station Record.	Experiment Station Record.
Gazzetta chimica italiana.	Gazzetta chimica italiana.
Geological Magazine.	Geological Magazine.
Neues Jahrbuch für Mineralogie, Geologie und Paläontologie.	Neues Jahrbuch für Mineralogie, Geologie und Paläontologie.
Neues Jahrbuch für Mineralogie, Geologie und Paläontologie. Beilage-Band.	Neues Jahrbuch für Mineralogie, Geologie und Paläontologie. Beilage-Band.
Jahrbuch der Radioaktivität und Elektronik.	Jahrbuch der Radioaktivität und Elektronik.
Journal of the American Chemical Society.	Journal of the American Chemical Society.
Journal of Biological Chemistry, New York.	Journal of Biological Chemistry, New York.
Journal de Chimie physique.	Journal de Chimie physique.
Journal of Geology.	Journal of Geology.
Journal of Hygiene.	Journal of Hygiene.
Journal of the Institute of Brewing.	Journal of the Institute of Brewing.
Journal für Landwirtschaft.	Journal für Landwirtschaft.
Journal of Medical Research.	Journal of Medical Research.
Journal of Pathology and Bacteriology.	Journal of Pathology and Bacteriology.
Journal de Pharmacie et de Chimie.	Journal de Pharmacie et de Chimie.
Journal of Physical Chemistry.	Journal of Physical Chemistry.
Journal of Physiology.	Journal of Physiology.
Journal de Physique.	Journal de Physique.
Journal für praktische Chemie.	Journal für praktische Chemie.
Journal of the Royal Agricultural Society.	Journal of the Royal Agricultural Society.
Journal of the Royal Society of New South Wales.	Journal of the Royal Society of New South Wales.
Journal of the Physical and Chemical Society of Russia.	Journal of the Physical and Chemical Society of Russia.
Journal of the Society of Chemical Industry.	Journal of the Society of Chemical Industry.
Journal of the Society of Dyers and Colourists.	Journal of the Society of Dyers and Colourists.
Kongl. Svenska Vetenskaps-Akademiens Handlingar.	Kongl. Svenska Vetenskaps-Akademiens Handlingar.
The Lancet.	The Lancet.
Die landwirtschaftlichen Versuchs-Stationen.	Die landwirtschaftlichen Versuchs-Stationen.
Memorie della Reale Accademia delle Scienze di Torino.	Memorie della Reale Accademia delle Scienze di Torino.
Memoirs of the College of Science and Engineering Kyôto Imperial University.	Memoirs of the College of Science and Engineering Kyôto Imperial University.
Memoirs and Proceedings of the Manchester Literary and Philosophical Society.	Memoirs and Proceedings of the Manchester Literary and Philosophical Society.
Metallurgie.	Metallurgie.
Milchwirtschaftliches Zentralblatt.	Milchwirtschaftliches Zentralblatt.
Mineralogical Magazine and Journal of the Mineralogical Society.	Mineralogical Magazine and Journal of the Mineralogical Society.
Monatshefte für Chemie und verwandte Theile anderer Wissenschaften.	Monatshefte für Chemie und verwandte Theile anderer Wissenschaften.
Il Nuovo Cimento.	Il Nuovo Cimento.
Archiv für die gesammte Physiologie des Menschen und der Thiere.	Archiv für die gesammte Physiologie des Menschen und der Thiere.
Pharmaceutical Journal.	Pharmaceutical Journal.
Pharmaceutical Review.	Pharmaceutical Review.
Pharmaceutisch Weekblad.	Pharmaceutisch Weekblad.
Pharmazeutische Zeitung.	Pharmazeutische Zeitung.
Pharmazeutische Zentrallhalle.	Pharmazeutische Zentrallhalle.
Philippine Journal of Science.	Philippine Journal of Science.
Philosophical Magazine (The London, Edinburgh and Dublin).	Philosophical Magazine (The London, Edinburgh and Dublin).

JOURNALS FROM WHICH ABSTRACTS ARE MADE

ABBREVIATED TITLE.	JOURNAL.
<i>Phil. Trans.</i>	Philosophical Transactions of the Royal London.
<i>Physikal. Zeitsch.</i>	Physikalische Zeitschrift.
<i>Proc. Amer. Physiol. Soc.</i>	Proceedings of the American Physiological Society.
<i>Proc. Camb. Phil. Soc.</i>	Proceedings of the Cambridge Philosophical Society.
<i>Proc. K. Akad. Wetensch. Amsterdam.</i>	Koninklijke Akademie van Wetenschappen Amsterdam. Proceedings (English version).
<i>Proc. Phil. Soc. Glasgow</i>	Proceedings of the Glasgow Philosophical Society.
<i>Proc. Physiol. Soc.</i>	Proceedings of the Physiological Society.
<i>Proc. Roy. Soc.</i>	Proceedings of the Royal Society.
<i>Proc. Roy. Soc. Edin.</i>	Proceedings of the Royal Society of Edinburgh.
<i>Quart. J. exp. Physiol.</i>	Quarterly Journal of experimental Physiology.
<i>Quart. J. Geol. Soc.</i>	Quarterly Journal of the Geological Society.
<i>Rec. trav. chim.</i>	Recueil des travaux chimiques des Pays-Bas. Belgique.
<i>Rend. Accad. Sci. Fis. Mat. Napoli.</i>	Rendiconto dell' Accademia delle Scienze Matematiche-Napoli.
<i>Rev. de Metallurgie</i>	Revue de Metallurgie.
<i>Rev. intern. Falsif.</i>	Revue internationale des Falsifications.
<i>Sci. Proc. Roy. Dubl. Soc.</i>	Scientific Proceedings of the Royal Dublin Society.
<i>Sci. Trans. Roy. Dubl. Soc.</i>	Scientific Transactions of the Royal Dublin Society.
<i>Sitzungsber. K. Akad. Wiss. Berlin.</i>	Sitzungsberichte der Königlich Preussischen Akademie der Wissenschaften zu Berlin.
<i>Sitzungsber. K. Akad. München.</i>	Sitzungsberichte der königlich bayerischen Akademie der Wissenschaften zu München.
<i>Trans. Amer. Electrochem. Soc.</i>	Transactions of the American Electrochemical Society.
<i>Trans. Amer. Inst. Mining Eng.</i>	Transactions of the American Institute of Engineers.
<i>Trans. Faraday Soc.</i>	Transactions of the Faraday Society.
<i>Trans. Nova Scotia Inst. Sci.</i>	Transactions of the Nova Scotia Institute of Science.
<i>Trans. Path. Soc.</i>	Transactions of the Pathological Society.
<i>Trans. Roy. Soc. Canada</i>	Transactions of the Royal Society of Canada.
<i>Trans. Roy. Soc. Edin.</i>	Transactions of the Royal Society of Edinburgh.
<i>Trans. Roy. Irish Acad.</i>	Transactions of the Royal Irish Academy.
<i>Tsch. Min. Mitt.</i>	Tschermak's Mineralogische Mitteilungen.
<i>U.S.A. Dept. Agric. Bull.</i>	Bulletins of the Department of Agriculture.
<i>U.S.A. Dept. Agric. Rep.</i>	Reports of the Department of Agriculture.
<i>Verh. des deut. Naturforsch. Aerzte.</i>	Verhandlung der Gesellschaft deutscher Naturforscher und Aerzte.
<i>Wiss. Abhandl. Phys.-Tech. Reichsanstalt.</i>	Wissenschaftliche Abhandlungen der Physikalisch-Technischen Reichsanstalt.
<i>Zeitsch. anal. Chem.</i>	Zeitschrift für analytische Chemie.
<i>Zeitsch. angew. Chem.</i>	Zeitschrift für angewandte Chemie.
<i>Zeitsch. anorg. Chem.</i>	Zeitschrift für anorganische Chemie.
<i>Zeitsch. Biol.</i>	Zeitschrift für Biologie.
<i>Zeitsch. Chem. Ind. Kolloide.</i>	Zeitschrift für Chemie und Industrie der Kolloide.
<i>Zeitsch. Elektrochem.</i>	Zeitschrift für Elektrochemie.
<i>Zeitsch. Farb.-Ind.</i>	Zeitschrift für Farben-Industrie.
<i>Zeitsch. Kryst. Min.</i>	Zeitschrift für Kristallographie und Mineralogie.
<i>Zeitsch. Nahr. Genussm.</i>	Zeitschrift für Untersuchung der Nahrungsmittel und Genussmittel.
<i>Zeitsch. öffentl. Chem.</i>	Zeitschrift für öffentliche Chemie.
<i>Zeitsch. physikal. Chem.</i>	Zeitschrift für physikalische Chemie, Elektrochemie und Verwandtschaftslehre.
<i>Zeitsch. physiol. Chem.</i>	Hoppe-Seyler's Zeitschrift für physiologische Chemie.
<i>Zeitsch. prakt. Geol.</i>	Zeitschrift für praktische Geologie.
<i>Zeitsch. Ver. deut. Zuckerind.</i>	Zeitschrift des Vereins der deutschen Zuckerindustrie.
<i>Zeitsch. wiss. Photograph. Photophysik. Photochem.</i>	Zeitschrift für wissenschaftliche Photographie, Physik und Photochemie.
<i>Zeitsch. Zuckerind. Böhm.</i>	Zeitschrift für Zuckerindustrie in Böhmen.

